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A SHEAR BOND STRENGTH, MICROLEAKAGE AND LASER MICROSCOPIC STUDY OF TWO DENTAL COMPOMERS.

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December, 1999



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Declaration

I, the undersigned hereby declare that the work contained in this dissertation is my own original work and has not previously in its entirety or in part been submitted at any university for a degree.



Signed.

11th November 1999

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Summary

Purpose: To evaluate and compare the *in-vitro* shear bond strength and microleakage of two compomers with their adhesive systems and to examine the dentine-restorative interface under confocal scanning laser microscopy (CSLM). **Materials and Methods:** For shear bond strength (SBS) testing thirty non-carious human molars were used of which fifteen molars were restored with Dyract AP using Non-Rinse Conditioner (NRC) and Prime&Bond NT (PBNT) and fifteen were restored with F2000 and Scotchbond Multi-Purpose Plus (SBMP). For the microleakage evaluation cavity preparations were made on the facial surfaces of thirty non-carious premolars. These were then restored with the respective compomer system. The specimens were thermocycled, sectioned and examined for dye penetration. The dentine-restorative interface was examined through a confocal scanning laser microscope. The primers of the bonding agents were labelled with rhodamine B and the adhesive resins were labelled with fluorescein and examined under CSLM in fluorescent mode. **Results:** The mean SBS for PBNT and SBMP were 12.8 and 18.1 MPa, respectively. The microleakage scores showed Dyract with PBNT leaked on the dentine side in 13 of the 15 specimens examined. On the enamel side 2 of the 15 specimens showed microleakage. With F2000 and SBMP no microleakage was observed on either enamel or dentine sides. The CSLM images show clear resin tag and hybrid layer formation for both the materials examined, although SBMP showed deeper penetration into the dentine with longer resin tags. The length of the resin tags and thickness of the hybrid layer for PBNT was found to be approximately 10 μm and 2 μm ,

respectively. SBMP showed resin tags measuring about 100 μm while the hybrid layer measured about 5 μm . ***Conclusion:*** This study demonstrates that the acid-etch technique of SBMP with F2000 produces higher bond strength and no microleakage when compared to the self-etching/self-priming “non-rinse technique” of NRC with PBNT and Dyract.



Opsomming

Doel: Om *in-vitro* skeursterkte en mikrolekkasie van twee kompomere met hulle bindingsisteme te evalueer en te vergelyk en die dentien-herstellingsinterfase te bestudeer met 'n konfokale laser skandeermikroskoop (KLSM). **Materiale en Metode:** Vir skeursterkte is altesaam dertig nie-karieuse menslike molare gebruik. Vyftien molare is met Dyract AP, Non-Rinse Conditioner (NRC) en Prime&Bond NT (PB NT) herstel en 'n verdere vyftien is met F2000 en Scotchbond Multi-Purpose Plus (SBMP) herstel. Vir mikrolekkasie evaluasies is kaviteite op die fasiale oppervlak van dertig nie-karieuse premolare voorberei. Hulle is onderskeidelik met dié twee kompomere sisteme herstel. Die monsters is daarna termies gesirkuleer, gesny en mikroskopies ondersoek vir kleurstof binnedringing. Die dentien-herstellingsinterfase is met behulp van 'n konfokale laser skandeermikroskoop ondersoek. Die bindingsagentprimer is gemerk met rhodamien B en die hegtingsharse met fluoresensie. Albei is met behulp van 'n konfokale laser skandeermikroskoop se fluoreserende verstelling ondersoek. **Resultate:** Die gemiddelde skeursterkte vir PB NT en vir SBMP was 12.8 en 18.1 MPa, onderskeidelik. Die mikrolekkasie evaluasie het aangetoon dat Dyract met PBNT aan die dentien kant in 13 uit die 15 monsters wat ondersoek is, gelek het. Aan die glasuur kant het 2 uit die 15 monsters gelek. Met F2000 en SBMP kon geen mikrolekkasie aan die glasuur of dentien kant aangetoon word nie. Die konfokale laser skandeermikroskoop beelde het goed gevormde penne [tags] asook 'n hibriedlaag vir beide die materiale aangedui alhoewel die SBMP dieper binnedringing van die dentien met langer harspenne getoon het. Die lengte

van die harspenne en deursnit van die hibriedlaag vir PBNT was ongeveer $10 \mu\text{m}$ en $2 \mu\text{m}$ onderskeidelik. SBMP se harspenne het ongeveer $100 \mu\text{m}$ en die hibriedlaag $5 \mu\text{m}$ gemeet. **Gevolgtrekking:** Hierdie studie het aangedui dat die suurets metode van SBMP met F2000 'n baie hoër bindsterkte gee, met geen mikrolekkasie nie, in teenstelling met die self-ets/self-priming “non-rinse technique” van NRC met PBNT en Dyract.



This thesis is dedicated to both my parents
for their love, support and guidance.



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CHAPTER I

Introduction

One of the most essential physical properties of a dental restorative material is that it should retain its size and form and not be dislodged once it is placed and contoured into the cavity preparation. Dimensional changes occurring in some restorative materials following placement into a cavity have limited their status as being classified as a "permanent filling" material. These dimensional changes can manifest clinically as microleakage or can even lead to dislodgment of the restoration. Testing the adhesion of materials is important as this will help in evaluating any dimensional changes that may occur. Ideal adhesive restorative materials should produce high bond strengths and long lasting, perfectly adapted restorations around the cavity margins without any dimensional changes thereby preventing any microleakage or debonding.

New materials are continuously being manufactured and in recent times seem to be "flooding" the dental market. Dental practitioners need to keep abreast with not only these new materials but also with the latest technologies that are constantly being developed. One of the new groups of materials to be developed recently and enjoying great popularity among dental practitioners is the compomers. Compomers is a result of continued modification and improvement of dental materials with the glass-ionomer cements at one end of the dental material spectrum and composite resins at the other end

with compomers lying somewhere in between these two groups. These compomers are correctly termed polyacid-modified resin composites.

Many of the newer materials are often accompanied only by literature prepared by the manufacturer. For obvious reasons many manufacturers claim their products to be the best on the market and there are very few or at times no independent studies available to justify these claims. The need for independent research is important as these publications will enable clinicians to compare and evaluate new materials and not having to rely solely on manufacturer's reports which at times may have a financial slant to their claims.

In-vitro research is necessary as clinical trials of these new materials can extend over long periods. *In-vitro* testing of the adhesive properties and sealing ability of materials is important, as this may give some indication of the behaviour of these materials *in-vivo*. One of the most common methods of evaluating adhesion of restorative materials to dentine and enamel is by measuring the bond strength at the tooth-restorative interface and by testing the marginal seal of restorations in extracted teeth (Gordan *et al.*, 1997).

Considerable improvement in dentine adhesion has occurred in recent years. Current development has focused on simplifying the application of bonding agents by decreasing the time and steps required for placement. As a result many of the newer bonding agents combine the primer and adhesive into a single component or a self-etching/self-priming system is used. While these systems are convenient to use, the efficacy of many of the newer materials has not yet been established.

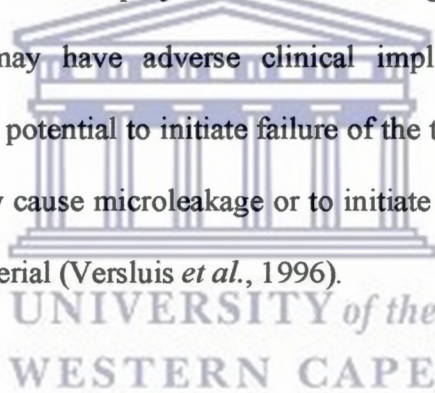
Therefore, the aim of this study was to evaluate shear bond strength and microleakage and to examine the micromorphological relationship between adhesive and dentine using confocal scanning laser microscopy, of two recently developed compomers and their adhesive systems *in-vitro*.



CHAPTER II

Review of literature

It is widely accepted that volumetric contraction occurs during the polymerization process of restorative materials (Crim, 1993; Versluis *et al.*, 1996). The conversion of monomer molecules into a polymer network is accompanied with closer packing of the molecules, which leads to contraction of the material. This reduction in volume is usually denoted as curing contraction or polymerization shrinkage. Stresses created by polymerization shrinkage may have adverse clinical implications. Polymerization contraction stresses have the potential to initiate failure of the tooth restorative interface (adhesive failure) which may cause microleakage or to initiate microcracking (cohesive failure) of the restorative material (Versluis *et al.*, 1996).



Microleakage

Polymerization shrinkage has been implicated in marginal gap formation and resultant microleakage (Crim, 1993). Microleakage may be defined as the passage of bacteria, fluid, molecules or ions between a cavity wall and the restorative material applied to it (Kidd, 1976).

Microleakage is a biological phenomenon with several clinical implications like:

- Recurrent caries.
- Tooth discolouration around the restoration.

- Hypersensitivity of the restored tooth.
- Pulpal inflammation. An *in-vivo* study by Cox (1994), in which combined culture and staining techniques showed that oral bacteria invade the restorative interface and penetrate the tubules causing pulpal inflammation and necrosis.
- Hastening the breakdown of certain types of restorative materials (Cox, 1994).

The prevention of microleakage depends largely on the maintenance of the seal between the restoration and the tooth structure. The adhesive restorative system must produce strong bonds to the cavity walls to be able to resist the stresses generated during polymerization shrinkage of the restorative resin as well as any other forces that may occur during normal occlusal functions.



Adhesion to tooth structure

It was Michael Buonocore in 1955 who first focused the attention of the profession to adhesion of restorative materials to tooth structure. Buonocore's procedure utilized phosphoric acid to etch the enamel, which then altered the surface morphology to create microporosity on the enamel surface. Resin materials were then bonded to this surface primarily through micromechanical adhesion. For many years, treatment time for acid conditioning of enamel was 60 seconds. Studies now indicate that treatment time can be reduced to 15 seconds. When compared to a 60-second conditioning time, a 15-second treatment time has been shown to create similar morphological changes in the enamel.

Also there is no significant difference in the bond strengths between these two treatment times (Barkmeier & Cooley, 1992). A 15-30 second treatment time with 30%-37% phosphoric acid is now widely used for etching enamel and the subsequent application of adhesive forms 2-5 μm resin tags into the etched enamel surface (Prati, 1998).

Bonding adhesive resins to acid etched enamel is now regarded reliable and reproducible. Strong enamel bonds require acids to clean the enamel surface, remove enamel smear layer and roughen the surface by selective removal of enamel rod material. Rinsing with water ensures the removal of solubilised calcium and phosphate and thorough drying is crucial to good enamel bonds (Pashley, 1991).

The success of acid etching enamel has led researchers to using phosphoric acid on dentine in efforts to obtain adhesion to this substrate. However, unlike enamel bonds, bonding to dentine proved more complicated and far more challenging. The heterogeneous structure of dentine, its vitality, its limited potential for microporosities, its high permeability to molecular flow and bacterial invasion and its wet surface all complicate the bonding of a resin to this hydrophilic surface (Van Meerbeek *et al.*, 1992). Dentine contains about 30% protein (mainly collagen). It is a composite of apatite crystal "filler" embedded in the collagen matrix. The crystalline phase is about 50% and water making about 20% (table 1).

Constituents	Mineralized	Demineralized	Resin-Infiltrated
Mineral	50	0	0
Collagen	30	30	30
Water	20	70	0-40
Resin	0	0	30-70

Table 1. Composition of dentine (%) as a bonding substrate during various stages in bonding procedures (Pashley et al., 1994).

Dentine is a living tissue and a vital part of the tooth structure in direct communication with the pulp. It is permeated by a system of tubules that originate at the pulp chamber and terminate in a branching network near the dentino-enamel and dentino-cemental junction (Pashley, 1991). The tubule lumen is lined by a highly mineralized cuff called peritubular dentine. The tubules are separated by intertubular dentine composed of a matrix of collagen reinforced by apatite. The fact that peritubular dentine is more highly mineralized than the intertubular dentine would mean that etching of the dentinal surface will demineralize the peritubular dentine more than the intertubular dentine thereby opening the tubules (Marshall et al., 1997).

Tubule density and orientation vary from location to location. Tubule density is lowest at the dentino-enamel junction and highest at the junction of the pulp chamber. Tubules occupy about 22% of the surface area near the pulp and about 1% near the enamel. This

difference is not only reflected in the depth of the tooth structure, but also in different parts of the tooth (figure 1). Proximal dentine is more permeable than occlusal dentine and coronal dentine is more permeable than root dentine (Pashley, 1991). This regional variation would affect the permeability of the adhesives into the tubules. Bonding to dentine is further complicated by the fact that dentine is modified by physiological, aging and disease processes to create different forms of dentine (Marshall *et al.*, 1997).

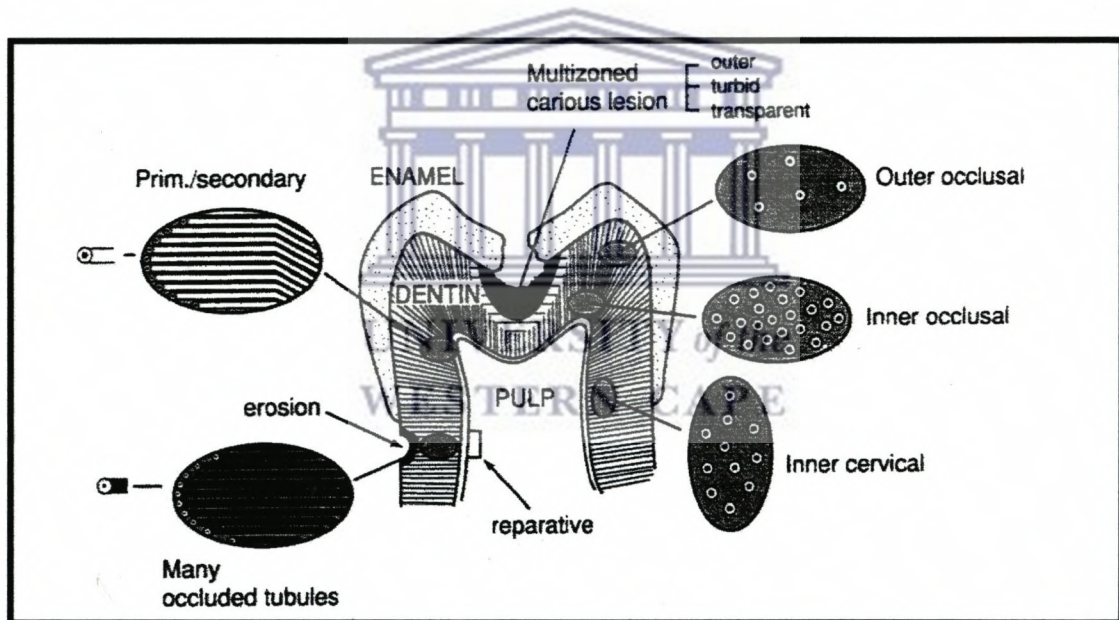


Figure 1. Schematic diagram of dentine structural variations with intratooth location, formation of secondary dentine and altered forms of dentine (Marshall *et al.*, 1997).

Bonding to dentine

Initial attempts to bond directly to etched dentine was met with limited success. Also concerns regarding adverse pulpal effects has refocused research on developing chemical adhesion to dentine (Barkmeier & Cooley, 1992). Initially, adhesion by coupling agents like NPG-GMA (N-phenylglycine glycidylmethacrylate) was utilized for bonding to dentine. Coupling agents utilizing the concept of hydrophobic and hydrophilic groups were the monomers based on phosphates and phosphonates. The hydrophilic phosphate is thought to interact with the calcium ions of the dentine (figure 2).

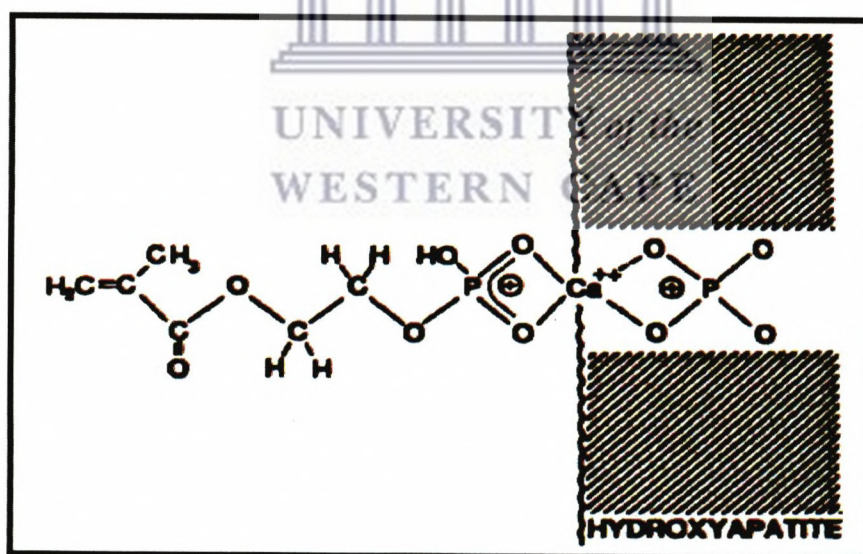


Figure 2. Bonding of a phosphate-based coupling agent to hydroxyapatite (Ruyter, 1992)

The so-called first and second generation dentine adhesives utilized phosphate bonding systems but yielded low bond strengths of about 2-7 MPa (Barkmeier & Cooley, 1992). These low bond strengths were too weak to counteract the polymerization shrinkage of composite resin. The materials separated from the margins forming marginal gaps leading to microleakage (Swift, 1998).

The next group or as was commonly termed third generation adhesives, differed from its predecessors in that they modified the dentine surface prior to the application of the resin. These adhesive systems used a conditioning step on dentine in conjunction with a bonding agent. The conditioning agents either modified or removed the smear layer and subsequently interacted with the superficial dentine. The initial oxalate system utilized a dentine conditioner of 2.5% nitric acid in combination with ferric oxalate. In the Gluma system EDTA (ethylene-diamine-tetra-acetic acid) was used as a dentine conditioner and HEMA (hydroxyethyl methacrylate) and glutaraldehyde as a dentine primer to graft the resin to the organic (collagen) component of dentine. Adhesion by grafting reactions was employed in these systems (Barkmeier & Cooley, 1992). Possible bonding sites of the collagen molecule include hydroxyl, carboxyl, amino and amido groups as shown in figure 3 (Asmussen & Uno, 1992). The glutaraldehyde would react with amino and amido groups in collagen and form charged N-(hydroxymethyl)-collagen compounds, which in turn would react with the OH group of the HEMA molecule (Burke & McCaughey, 1995).

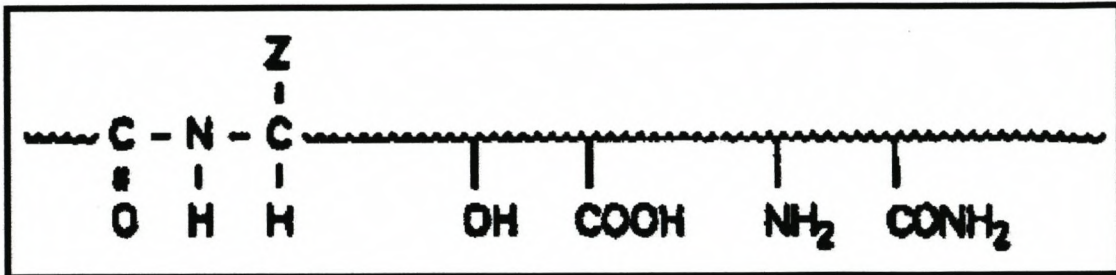


Figure 3. Bonding sites available on the dentinal collagen molecule – constituent Z may vary depending on the dentine bonding agent (Asmussen & Uno, 1992)

Again low bond strengths of about 10 MPa were reported with these systems (Barkmeier & Cooley, 1992). Although these dentine adhesives were more effective than their predecessors in reducing microleakage they did not eliminate marginal leakage.

The hallmarks of the fourth generation materials are the concepts of total etching and moist dentinal bonding. The bonding mechanism of the fourth generation adhesive systems is a three-step process: (1) condition, (2) prime, and (3) bond.

Conditioning or acid etching removes the smear layer, decalcifies the intertubular and peritubular dentine, opens the dentinal tubules and increases dentine permeability. Removal of hydroxyapatite crystals leaves a collagen meshwork on the dentine surface (Swift, 1998). With these systems bond strengths have dramatically increased (>15 MPa).

Many of the earlier systems developed relatively weak bonds probably because of their inability to penetrate the dentine surface effectively. Current approaches appear to depend on effective penetration of the partially demineralized dentine (Marshall *et al.*, 1997).

Dentine permeability

A key element for adhesion is the intimate association of adhesive and substrate. The permeability of dentine to adhesive agents is of crucial importance in obtaining good dentinal bonding. During bonding the adhesive resin infiltrates vertically via any porosities in the intertubular dentine created by acid etching (figure 4). Pashley & Carvalho (1997) termed this infiltration *intertubular permeability*. The channels formed during demineralization allow the resin to diffuse and infiltrate into the demineralized dentine and envelop the collagen fibers. The adhesive in ways substitute the apatite phase of the dentine and creates a new layer of resin in a collagen matrix termed “hybrid layer” by Nakabayashi *et al.* (1992). Hybridization is neither resin nor collagen but represents a zone of resin diffusion into the intertubular dentine. This results in micromechanical entrapment of the resin within the collagen fibrils and may be responsible for durable bonds to dentine (Nakabayashi *et al.*, 1992). However, Gwinnett *et al.* (1995) indicated that the reinforced or hybridized collagen network does not contribute quantitatively to the bond strength. When the smear layer covered dentine surface is acid etched and subsequently treated with collagenase, a clean, porous substrate is produced. It is speculated that this tissue is attacked by the acidic primers, slightly etching it and permitting shallow resin penetration sufficient to produce high bond strengths (Eick *et al.*, 1997). Deeper penetration of the resin into the dentine tubules and anastomosis was observed when the collagen was removed suggesting that the removal of this layer allows for better resin penetration into the dentine (Vargas *et al.*, 1997a).

The deeper the acidic conditioners penetrate the dentine during acid etching, the deeper the adhesive monomer must penetrate or permeate the demineralized dentine. Ideally, the resin should penetrate the very base of the demineralized dentine, replace the water and completely envelop the collagen fibrils. If they do not, there will be an area of demineralized collagen fibres that would not be enveloped by resin. This “non-hybridized collagen” may provide an area with a potential for slow hydrolysis of any demineralized dentine and can jeopardize the durability of a good bond (Pashley *et al.*, 1994). Stable bonds can be formed if resins penetrate less deeply but more uniformly rather than deeper but spotty and non-uniform infiltration into the demineralized zone (Pashley & Carvalho, 1997).

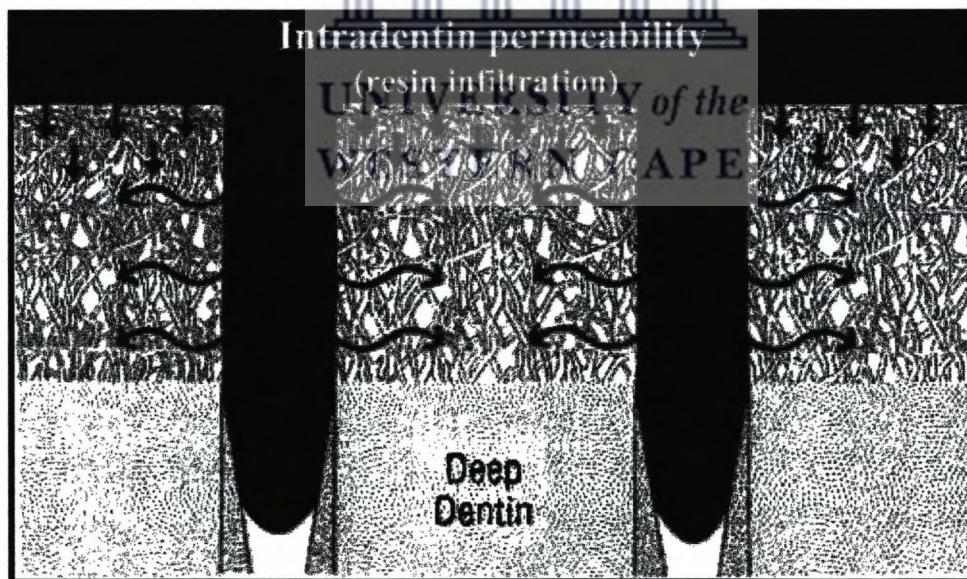


Figure 4. Resin infiltration into dentine (Pashley *et al.*, 1994)

Role of resin tags

Besides the infiltration of resin through the porous intertubular dentine (intertubular permeation), a second type of penetration of resin occurs. This infiltration is termed *intratubular permeation* and occurs with the movement of resin into the tubule lumina. The resin penetrates the dentinal tubules to form long resin tags which if bonded to the walls will seal the tubules and contribute to resin retention. Because the dentinal tubules converge towards the pulp, resin will penetrate the tubules in different directions converging to the pulp thus providing non-parallel retention (Pashley & Carvalho, 1997). However, longer resin tags does not necessarily imply stronger bond strengths.

Following acid etching of dentine the tubule orifices become enlarged and funnel shaped. Etching removes the mineral of about 3-4 μm of the top of the dentinal tubules and it exposes the collagen network that communicates with the adjacent collagen in the intertubular dentine. This would permit lateral or horizontal permeation of resin and occurs via porosities on the walls of the dentinal tubules. These form lateral sub-micron resin tags. The resin infiltration occurring adjacent to the orifices and enveloping the collagen fibrils would lead to hybridization at the openings of the tubules. This phenomenon has been termed “lateral tubule hybridization” (Van Meerbeek *et al.*, 1998). Polymerization of resin in the lateral canals produces a three dimensional network of interconnected adhesive tags that may further contribute to a stronger dentine/resin bond (Chappell *et al.*, 1994).

The short time permitted before light curing limits the distance the resin can permeate the demineralized dentine. There may be a subsurface zone that is below the hybrid layer that may not be infiltrated by resin leaving an area of demineralized dentine susceptible to hydrolysis. This may permit leakage which Sano *et al.* (1995) termed “nanoleakage” as this leakage may occur below the hybrid layer and to distinguish it from classical microleakage. Sano *et al.* (1995) using silver nitrate showed in spite of the absence of gaps around the marginal walls of the cavity the presence of small amounts of silver nitrate below the hybrid layer suggesting leakage other than the traditional microleakage around the walls could occur. The leakage pathway through a porous zone at the hybrid-adhesive interface in the absence of gap formation is not microleakage in the classic sense, hence the term “nanoleakage”. This leakage occurs in nanometer (20-100 nm) sized channels (Sano *et al.*, 1995). The clinical significance of nanoleakage is still not clear, but it indirectly reflects the inability of adhesive systems to completely fill the demineralized zone (Pashley & Carvalho, 1997).

It has been hypothesized that resin tags that are longer than 2-3 μm may not contribute to retentive strength any more than those that are 2-3 μm long (Pashley & Carvalho, 1997). Hybridization occurs laterally at this point into the exposed intertubular dentine, but any deeper extensions of the resin tags lie passively within the tubules and do not appear to contribute to the attachment mechanism (Walshaw & McComb, 1996). Resin tag penetration in the open tubules along with lateral tubule hybridization is favourable in hermetically sealing the pulpo-dentinal complex against microleakage. Resin tags should

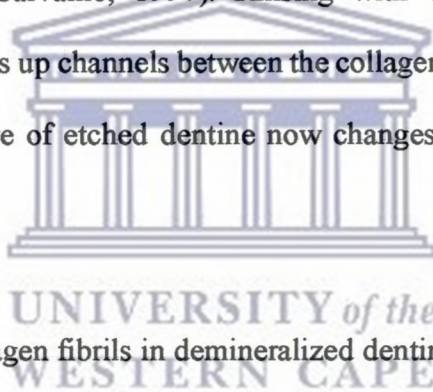
be considered as part of the hybrid layer rather than separate entities in the evaluation of effective dentine bonds (Titley *et al.*, 1995).

Resin tag formation is important as they act as anchors in the dentine and thereby increasing the bond strength of the restoration to dentine. Resin tags play an important role in preventing shrinkage, by functioning as anchor materials and may prevent leakage by sealing the dentine tubules. Collagen morphology around the tubules after acid etching may influence the morphology of the resin tags. The greater the diameter of the tubules, the larger the diameter of the resin tags. The larger the tag diameter, the higher the retentive value. Collapse of the collagen around the tubules may prevent resin tag formation or may produce very fine tags with low retentive value (Chersoni *et al.*, 1997).

In most current-generation bonding systems the bonding forces between the resin tags and the tubule walls exceed the cohesive strength of the resin tags. Thus, during debonding cohesive fractures of the resin tags at the dentine surface will occur. Therefore, besides contributing to the overall strength of the resin-dentine bond, the major role of the resin tags is to effectively plug the tubule orifices, reducing the chance for increased permeability and potential pulpal irritation (Eick *et al.*, 1997).

Role of water

Water is intimately involved in dentine bonding. The water present during bonding procedures comes from several sources such as dentinal fluid, atmospheric water, rinsing procedures and from the adhesive solutions as well. In the past, water was considered a contaminant which would compete against resin bonding (Pashley & Carvalho, 1997). When dentine is acid etched (or “conditioned”) with 37% phosphoric acid for 15 seconds the smear layer and smear plugs are removed and the top 3-7 μm of sound dentine is demineralized (Pashley & Carvalho, 1997). Rinsing with water then removes the solubilized material and opens up channels between the collagen fibrils. Following water rinsing the chemical structure of etched dentine now changes to 70% water and 30% collagen (table 1).



The spaces between the collagen fibrils in demineralized dentine are very small (0.01 to 0.03 μm wide) and are essentially maintained open by water (Pashley *et al.*, 1994). Water is responsible for keeping the collagen in an expanded state thereby preserving the porosity necessary for permeation of resins. If this dentine is allowed to dry or if air is blown onto demineralized dentine the water content will dramatically decrease. This will cause shrinkage or collapse of the collagen which in turn causes the width of the spaces or channels between the collagen fibers to shrink (Pashley *et al.*, 1994). Marshall *et al.* (1997) reported that demineralized dentine underwent dramatic collapse on drying until a dense collagen mat remained, and such collapse was so rapid that changes were difficult

to follow with atomic force microscopy. As these are the very spaces between the collagen fibers through which the resin must diffuse in order to infiltrate the demineralized dentine, the permeability of the resin will be greatly reduced.

In order to avoid this collapse of dentine by drying, Gwinnett & Kanca (1992) and Gwinnett (1992) advocated “wet bonding”. Both these authors have demonstrated that bonding to dentine under moist conditions could result in higher bond strengths than bonding to dry dentine (figure 5).

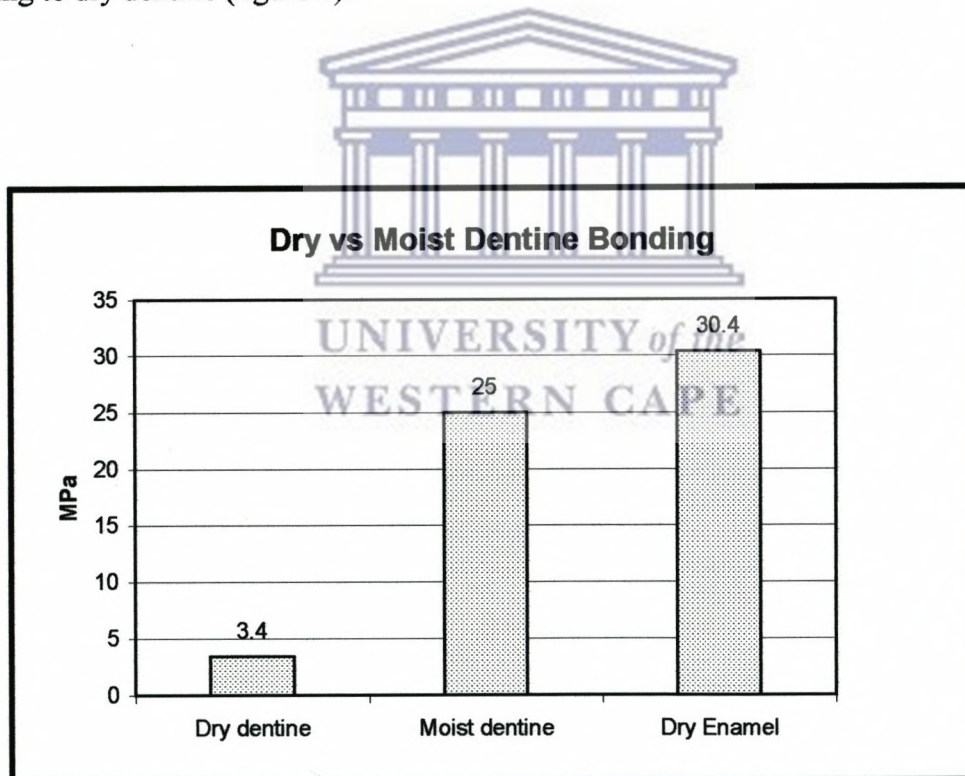


Figure 5. Graphical display of shear bond strengths of All-Etch/All-Bond to dry and moist dentine and dry enamel (Kanca, 1992).

By wet bonding it is meant, following etching and rinsing, the dentine must not be dehydrated or desiccated. These authors suggest blot drying the dentine surface with a damp cotton pellet.

However, the amount of water remaining is critical as excess water will result in poor bond strengths (Pashley *et al.*, 1994). Any increase in water concentration fills the surface and subsurface with water which makes it more difficult for hydrophilic monomers to displace this water. The water can also dilute the concentration of the hydrophilic monomers (Pashley & Carvalho, 1997). Hence, the term “moist bonding” is preferred to “wet bonding”. Marshall *et al.* (1997) observed that on rehydration of the dry dentine surface, the collapsed collagen recovered nearly all of its height *i.e.* the shrinkage of the demineralized collagen matrix appears to be reversible.

Adhesive agents applied to moist dentine must still physically replace the water to infiltrate into the spaces between the exposed collagen fibrils. Primers in the adhesive systems are adhesion-promoting agents that contain monomers. Hydroxyethyl-methacrylate (HEMA), the most commonly used wetting agent acts as a bifunctional resin. These monomers have hydrophilic properties (hydroxyethyl component) which have an affinity for exposed collagen fibril arrangement and hydrophobic properties (methacrylate component) for co-polymerizing with the adhesive resin (Van Meerbeek *et al.*, 1998). The monomers are often dissolved in organic solvents such as acetone or ethanol. Organic solvents in the primer remove water in the demineralized zone by

diffusion. When the primer is applied to the moist demineralized dentine, the water diffuses from the wet dentine while the acetone diffuses into the dentine matrix. The primer monomers dissolved in the acetone diffuse into the spaces previously occupied by water. The water is gradually lost as the solvents and resin monomers occupy the spaces around the collagen fibrils. In this way there is less shrinkage or collapse of the collagen network (Pashley & Carvalho, 1997). Further, the addition of acetone causes a decrease in the surface tension of the mixture.

For strong adhesion to take place there must be good wetting and intimate contact between the adhesive and the substrate. Liquids “wet” a surface when they spread across it readily rather than remaining beaded on the surface. The critical surface tension represents the surface tension of a liquid that allows a drop to spread across the surface so that its contact angle is zero. It is desirable that the adhesive liquid has a surface tension equal to or less than the critical surface tension for the solid substrate. Unlike enamel, where the surface energy increases following acid etching, the surface energy of dentine decreases, probably due to the higher concentration of collagen. If adhesive resins, which generally have higher surface tensions, were applied directly to etched dentine, the mismatch between their surface energy and that of dentine would not permit effective wetting to occur. Primers generally have a surface tension that is less than the free surface energy of the acid-etched dentine (Eick *et al.*, 1997). This will permit adequate wetting and good contact of the primer to the dentine substrate.

Role of smear layer

Prior to acid etching both the tubules and the intertubular dentine are covered by a smear layer whenever dentine is cut with rotary or hand instruments (figure 6). These particles consist of pieces of dentine matrix *i.e.* the chemical composition of the smear layer is the same as that of the dentine from which it was created.

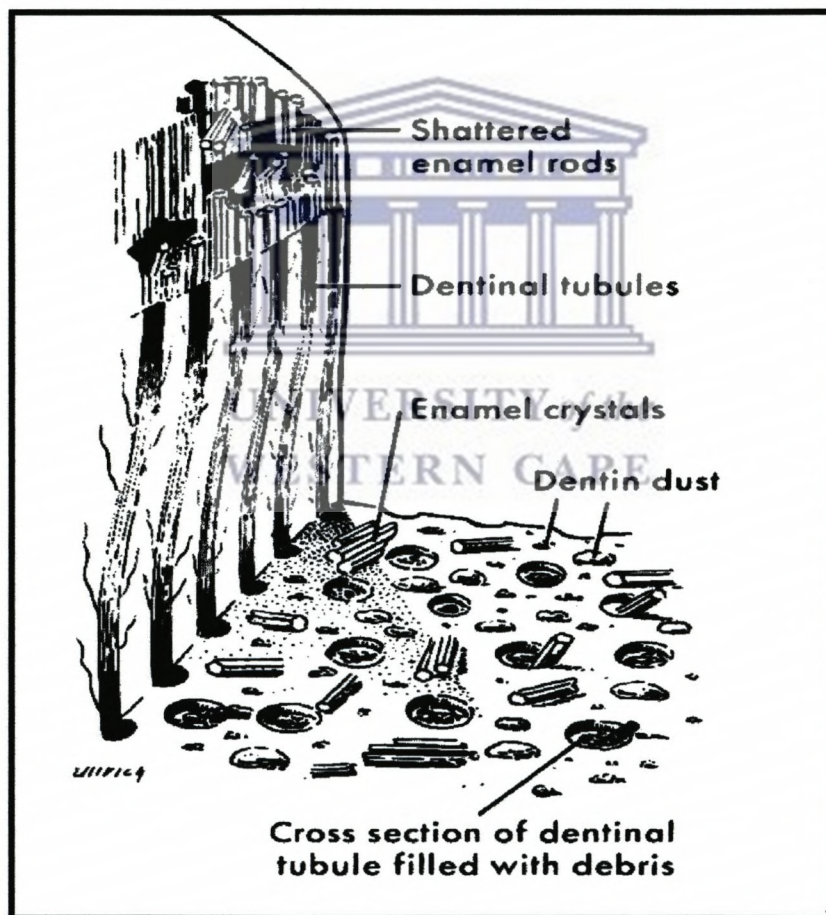


Figure 6. Schematic diagram showing smear layer on dentine surface (Tylman & Malone, 1978).

Thus, the smear layer in superficial, normal dentine may have a composition close to that of intertubular dentine, whereas the composition of the smear layer in deep dentine would reflect its lesser degree of mineralization. Similarly smear layers created on caries affected tissue may contain collagen that has been denatured by the action of proteolytic enzymes from cariogenic bacteria (Eick *et al.*, 1997). Smear layer particles vary from about 0.05 to 10 μm in size although they seldom stack more than 1 μm thick (Pashley & Carvalho, 1997).

Acid etching the smear layer only solubilizes the mineral component resulting in the formation of a smear layer of collagen particles. If this layer is air-dried the collagen will lose its water support and collapse causing it to be less permeable to resin monomers (Pashley & Carvalho, 1997). This is called the “collagen smear layer” (Pashley *et al.*, 1994) and it occupies the interface between the adhesive resin and the underlying demineralized dentine. This can inhibit the infiltration of the resin into the demineralized dentine.

With the new self-etching/self-priming systems the smear layer is used as a bonding substrate since the self-etching/self-primer is not rinsed off from the surface and the demineralized smear layer may be incorporated into the hybrid layer (Eick *et al.*, 1997). These newer bonding systems do not have a separate acid-etch component and no rinsing is required. The primers in these systems are acidic enough to demineralize the smear layer and the top of the underlying dentine surface. As they etch, they also infiltrate the

exposed collagen with hydrophilic monomers, which then co-polymerizes with the subsequently placed adhesive resin. The concept of an acidic primer is attractive, because in theory, this system simultaneously infiltrates the collagen fibres as it decalcifies the inorganic component to the same depth in dentine. This should minimize voids, since there would not exist a region of demineralized dentine that was not encapsulated by the resin primer (Gordan *et al.*, 1997).

Since the primed surfaces are not rinsed with water, the solubilized mineral will reprecipitate within the diffusion channels created by the acidic primers (Eick *et al.*, 1997). The infiltration of acidic resins through the smear layer into the underlying dentine matrix creates a special type of hybrid layer which contains an upper smear layer zone and a lower demineralized dentine zone infiltrated by the acidic primer (Gordan *et al.*, 1997). The presence of the smear layer greatly reduces the transdental permeation of resin and may account for narrower hybrid layer in these self-etching/self-priming systems. However, there is no correlation between the thickness of the hybrid layer and bond strength (Yoshiyama *et al.*, 1998). In deeper dentine the thickness of the hybrid layer was found to be much greater than in superficial dentine. This may be due to the fact that acid etching is more rapid in deep dentine because the acids penetrate the dentine tubules faster than they can penetrate the intertubular dentine. Also, resin infiltration into deep dentine may be more efficient than in superficial dentine because there are more tubules per unit area in deep dentine. Bond strengths were found to be

higher in superficial dentine with narrower hybrid layer than in deep dentine with thicker hybrid layer (Yoshiyama *et al.*, 1995).

Resins can only reach the dentinal tubules and intertubular dentine when they are able to diffuse through the smear layer. The acidic primers in these systems must be able to permeate through the water filled channels between the particles of the smear layer (Pashley & Carvalho, 1997). If the resin can infiltrate as little as 0.1-0.2 μm into superficial dentine, there may be enough retention to produce a bond strength of about 20 MPa (Yoshiyama *et al.*, 1998).

Although the smear layer was considered a limiting factor with earlier bonding agents, it is now regarded as a legitimate bonding substrate (Pashley & Carvalho, 1997).

Both intratubular and intertubular permeation is critical to achieve optimal bond strength and sealing of dentine tubules. In superficial dentine which contains fewer tubules per unit area than deeper dentine, permeation of resins into intertubular dentine will be responsible for most of the bond strength. In deeper dentine, dentinal tubules are more abundant and, intratubular permeation of resins to form resin tags will probably be responsible for a large fraction of the retention and sealing (Pashley & Carvalho, 1997).

In the absence of hybrid layer and resin tag formation on non-etched dentine surface, adhesion to intertubular dentine is probably the bonding mechanism available (Yoshiyama *et al.*, 1995).

Kanca & Sandrik (1998) suggested that the demineralized collagen in the hybrid layer does not contribute to the quality or strength of the adhesion. Armstrong *et al.* (1998) found that the interfacial fracture toughness is not significantly different with or without the micro-mechanical bond of the hybrid layer. Vargas *et al.* (1997a) also supports the theory that the collagen layer may not be crucial to the mechanism of adhesion between resin and dentine. In fact, this unsupported collagen layer may inhibit penetration of resin into the dentine substrate and undermine the long-term durability of the resin to dentine bond. Durable bonds may be facilitated by collagen layer removal, allowing more complete resin diffusion into the partially demineralized dentine (Vargas *et al.*, 1997a).

The optimal dentinal surface pretreatment has yet to be determined for adhesive integrity of resin to dentine. Although constant innovation has improved the performance of dentine adhesives, the true mechanism of resin adhesion to dentine is still not clear (Kanca & Sandrik, 1998).

Mount (1994) suggested, with the introduction of glass-ionomer cements, that the problem of adhesion to tooth structure might be solved.

Glass-Ionomer Cements

Since its introduction by Wilson and Kent in 1969, glass-ionomer cements (GIC) have become increasingly popular in restorative dentistry (Puckett *et al.*, 1995). Glass-ionomer cement is defined as a cement that consists of a basic glass and an acidic polymer which sets by an acid-base reaction between these components (McLean *et al.*, 1994). The acid is a polymer or copolymer of alkenoic acids such as acrylic acid, maleic acid, and itaconic acid.

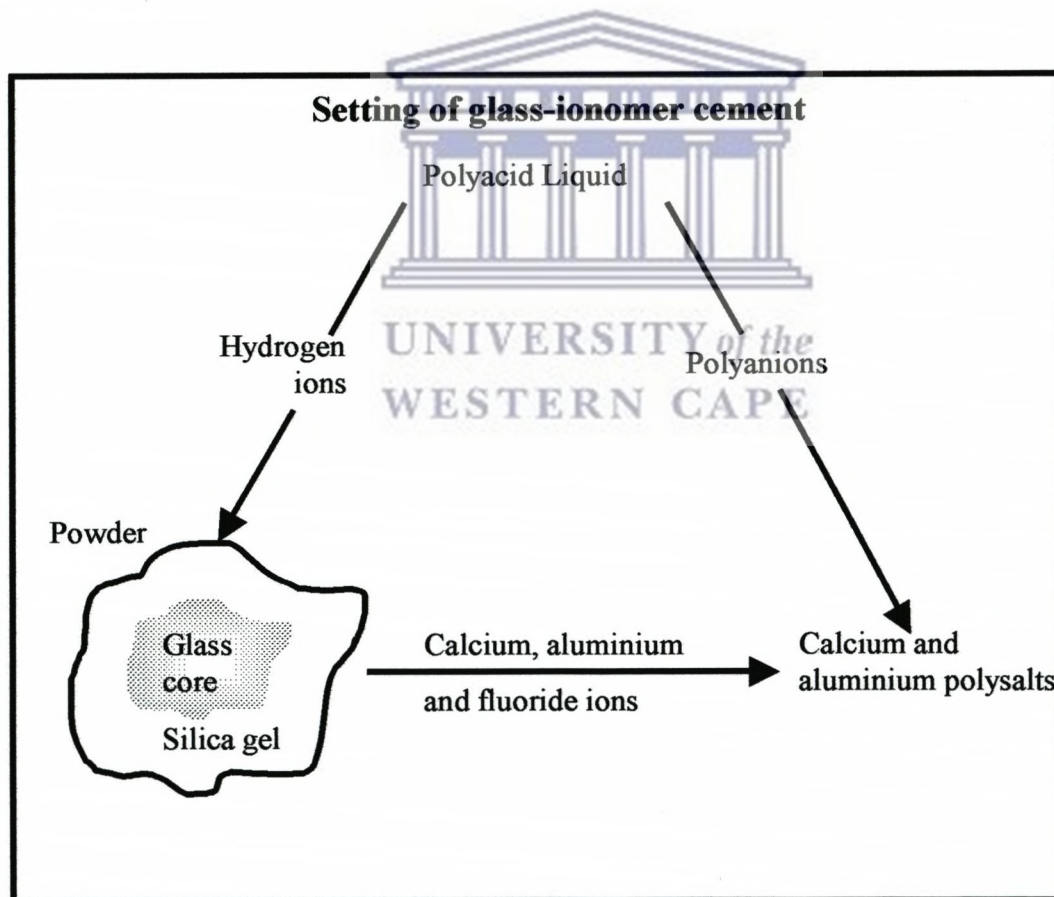


Figure 7. Cement forming reaction of glass-ionomer cements (Wilson & McLean 1988).

The base component is a calcium aluminosilicate glass containing fluoride (Peutzfeldt, 1996). This glass is acid decomposable and usually ground to a fine powder (McLean *et al.*, 1994). Thus, glass-ionomer cements comprises an aqueous solution of an acidic polymer and a reactive, acid soluble glass (figure 7).

When the powder and liquid are mixed together, the glass starts to dissolve in the acidic solution thereby releasing fluoride ions, metal ions and silica. The first stage of the reaction is the ionization of the carboxyl radicle, COOH to COO^- (carboxylate ion) and H^+ . The H^+ ion then acts on the surface of the glass particles (Katsuyama *et al.*, 1993). The hydrated protons from the liquid attack the surface of the glass particles with the release of Al^{+3} and Ca^{+2} cations. When this happens, the aluminosilicate glass is broken down into silicic acid (H_4SiO_4). The silicic acid slowly causes a condensation reaction by means of its OH specie, and becomes a porous gel having a rope-like structure. Metallic salt bridges are formed between polyacrylic acid ions in the liquid, and the cross-linked gel matrix is formed as the initial set of the mix develops causing it to harden. The unreacted glass particles become surrounded with gel. During the initial setting reaction, calcium ions are more rapidly bound to the polyacrylate chains, whereas a more prolonged and permanent set occurs as the trivalent aluminum ion-exchange enters the reaction and strengthens the cross-linking effect. This chemical setting reaction is illustrated in figure 8.

The Ca^{+2} and Al^{+3} crosslink with two or three COO^- ions of the polyacrylic acid chain and this becomes a gel. This gel is hydrated and its strength increases as the hydrated gel structure progresses (Katsuyama *et al.*, 1993).

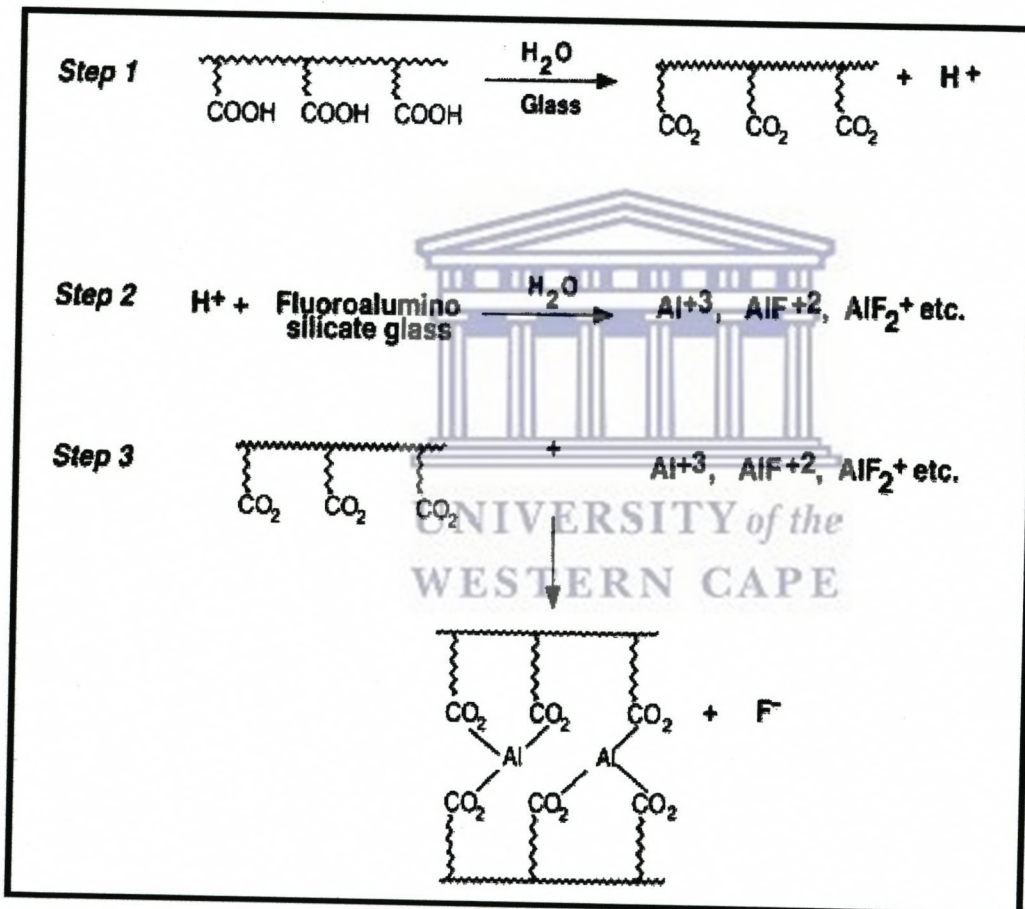


Figure 8. Chemical setting reaction of glass-ionomer cement (Vitremer Technical Profile, 1992).

When the acid attacks the surface of the powder particles, releasing calcium and aluminum ions, a diffusion-based adhesion develops between the powder and the liquid. A similar diffusion-based adhesion occurs on the surface of the tooth because phosphate ions are displaced by the polyalkenoic acid as it attacks and penetrates both the dentine and enamel (Mount, 1994). Each phosphate ion takes with it a calcium ion from the tooth structure to maintain the electrolytic balance at the interface.

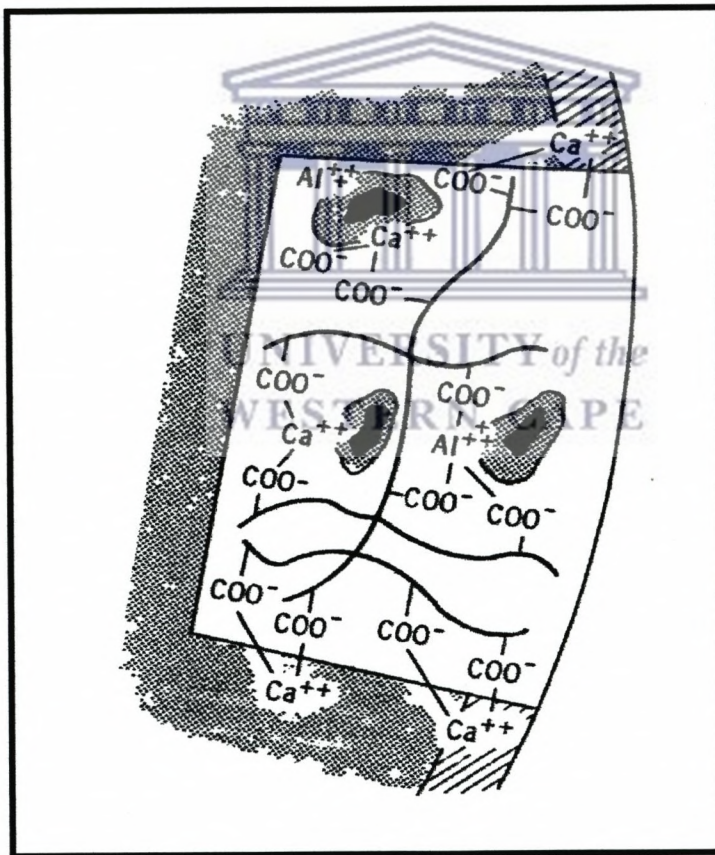


Figure 9. Adhesion of glass-ionomer cement to tooth structure (Phillips, 1991).

These ions will combine with the surface layer of the cement and form an intermediate layer that is firmly attached to the tooth structure (figure 9). The union is based initially on hydrogen bonding and, over time, matures into a stronger chemical bond of an ionic kind (Mount, 1999). Fluoride ions are not an essential part of the matrix formation and they are free to move both out of and back into the cement (Mount, 1994).

To obtain good adhesion between tooth and material via ionic exchange it is necessary to change the surface of the tooth structure from high energy to low energy to encourage total adaptation of the cement to the dentine. A ten second application of a 10% solution of polyacrylic acid will lower the surface energy so that the cement, which has a high surface energy will adapt and flow readily over the tooth. The polyacrylic acid has a double action in that it removes the smear layer and at the same time lowers the surface energy (Mount, 1994). Hence, the term "conditioning" has been used for this action. A further advantage of this particular acid is that it is part of the GIC system, so any remaining residue will not interfere with the setting reaction of the cement (Mount, 1999).

In the presence of this type of adhesion failure leading to loss of a restoration will be cohesive in the cement rather than adhesive between the two materials leaving a thin layer of ion-enriched material firmly attached to the tooth structure (Mount, 1999). Because of the favourable adhesion and fluoride releasing properties, GIC's has found widespread use as a restorative, lining and luting material. However, early versions of

this material had several disadvantages, including early dissolution and dehydration, early dislodgment, postoperative sensitivity and poor colour stability (Puckett *et al.*, 1995). Another disadvantage of the earlier versions of GIC's is the short working time and long setting time.

Resin-modified glass ionomer cements

To overcome the above shortcomings of conventional glass-ionomer cements and at the same time maintaining their clinical advantages, a new generation of cements was introduced. These cements are termed resin modified glass-ionomer cements (RM GIC). Hydroxyethyl methacrylate (HEMA) replaces some of the water component of the conventional glass-ionomer cement and the fundamental acid-base curing reaction is supplemented by a second curing process which is initiated by light (Sidhu & Watson, 1995). The acrylic acid chain is modified by grafting acrylate side groups onto the polyacid of the GICs and upon polymerization of the cement, the modified polyacid copolymerizes with the hydrophilic monomer *i.e.* HEMA (Peutzfeldt, 1996).

The light activated command set is available over the top of this reaction. The set material is due to the formation of a polymerization matrix on application of a light source while the acid-base reaction hardens and strengthens the matrix (Sidhu & Watson, 1995). Hence, the auto-setting acid-base reaction of conventional GICs is supplemented by the light initiated free radical reaction of the resins. This chemical setting reaction is illustrated in figure 10. The addition of a small quantity of light curing resin to

conventional GIC's has resulted in materials with significantly improved mechanical properties, increased bond strengths and considerably higher resistance to early moisture contact and desiccation (Van Dijken, 1996).

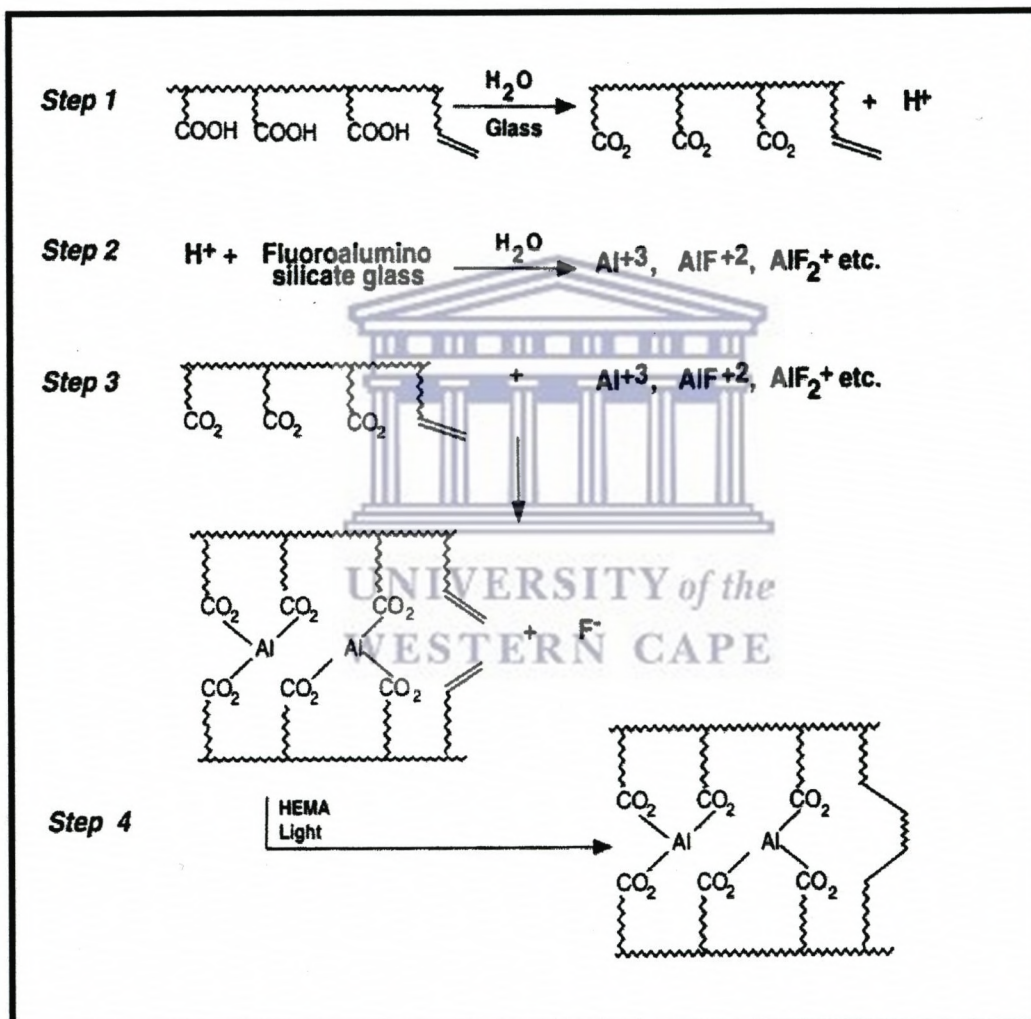
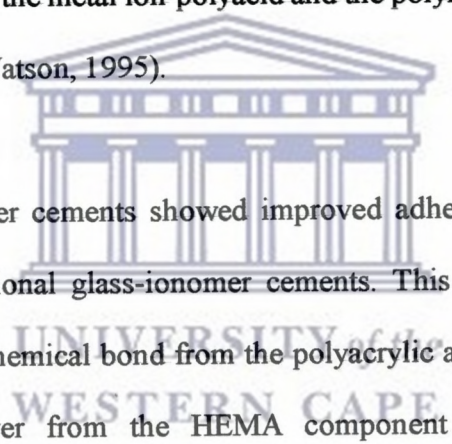


Figure 10. Chemical setting reaction of resin-modified glass ionomer cement (Vitremer Technical Profile, 1992).

The term dual cure has been suggested because these cements undergo the original acid-base reaction and there is light activation superimposed over this. As soon as the powder and liquid are combined, the acid-base reaction begins as usual and will continue in the same way over many weeks. However, a light activated command set is available over the top of this reaction which will immediately protect the acid-base reaction from the problems of water balance and stabilize the setting cement (Mount, 1994). The set cement is formed by two inter-penetrating matrices *i.e.* the ionic matrix from the acid-base cross-linking reaction of the metal ion-polyacid and the polymerization matrix of the monomer system (Sidhu & Watson, 1995).

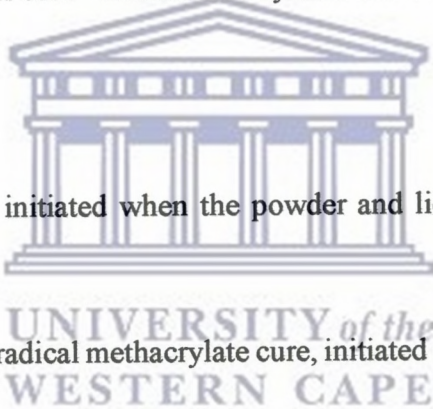


Resin modified glass-ionomer cements showed improved adhesion to tooth structure when compared to conventional glass-ionomer cements. This improved adhesion is probably caused by both a chemical bond from the polyacrylic acid component and the formation of a hybrid layer from the HEMA component (Van Dijken, 1996). Comparisons of shear bond strength of RM GICs to dentine with conventional glass-ionomers reveal the strength of the former is generally greater (Sidhu & Watson, 1995). Carvalho *et al.* (1995) using scanning electron microscope images revealed the formation of a hybrid-like layer with the same features observed with resin composites, suggesting the presence of micro-mechanical retention to dentine.

Polymerization shrinkage of the resin component in the resin modified glass-ionomer cements could theoretically cause such materials to exhibit greater microleakage than

conventional glass-ionomer cements. Any increase in microleakage with these materials could also be attributed to thermal expansion mismatch with the tooth structure. The thermal expansion of glass-ionomer cement is similar to that of tooth structure, while that of resin composite differs greatly (Yap *et al.*, 1995).

A further advancement in the chemical cure to ensure complete cure over time is called the “tricure” or ‘dark cure’ in the form of a commercial product Vitremer^b. The manufacturer refers to it as “tri-cure” because the system has the following three curing reactions:

- 
- Acid-base glass-ionomer, initiated when the powder and liquid are mixed and can proceed in the dark.
 - Photopolymerization free radical methacrylate cure, initiated when powder/liquid mix is exposed to light and occurs only when light penetrates.
 - Dark cure free radical methacrylate cure, initiated when powder and liquid are mixed and can proceed in the dark (Vitremer Technical Profile, 1992). This setting reaction is shown diagrammatically in figure 11.

However, this is not really a triple-cure mechanism, but a dual-cure mechanism in which one of the processes has two modes of initiation, *i.e.* curing of free radicals chemically and photochemically (Sidhu & Watson, 1995). The manufacturer claims that the

advantage of this system is that cure can be achieved beyond the depth of penetration of the curing light *i.e.* in bulk or core build up.

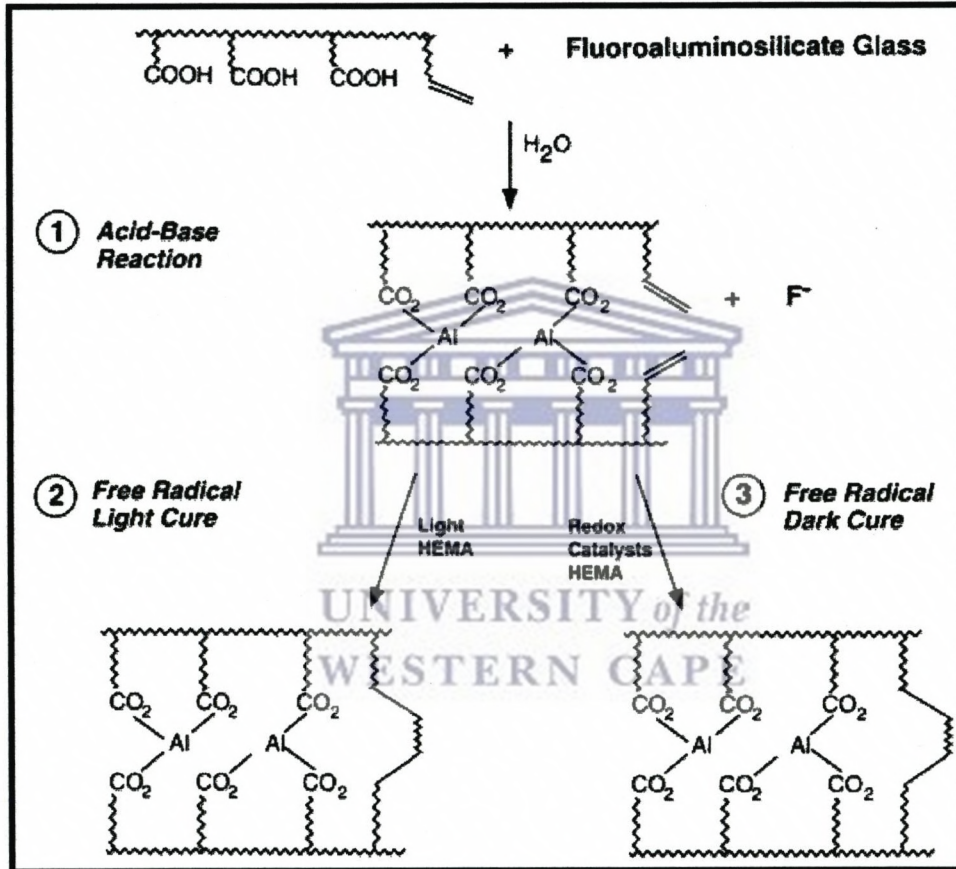


Figure 11. Setting reaction of Vitremer Tri-Cure Glass-Ionomer Cement (Vitremer Technical Profile, 1992).

Compomers

Following the trend to develop an ideal restorative material, a new class of materials was marketed in the nineties. These materials contain acid-decomposable glass and acidic polymerizable monomers substituting the polyalkenoic acid polymer. McLean *et al.* suggested the term polyacid-modified resin composite for these materials, but they became commonly known as compomers (Van Dijken, 1996).

The term compomers is now widely used to describe this water-free, single-component, light-cured composites consisting of polyacid-modified dimethacrylate monomers reinforced with strontium or barium aluminosilicate glass particles. In the presence of water, acid groups are expected to ionize and to interact with the basic glass components to produce a glass-ionomer core analog with anticariogenic properties due to fluoride release (Eliades *et al.*, 1998).

The word “compomer” was derived by combining parts of two words, COMPOSite and ionoMER and intended to suggest a combination of composite and glass-ionomer technology. Would this imply a combination of the properties of both these components? Ideally the material should combine the best properties of composites and glass-ionomer cements. Compomers have been reported to having the anticariogenicity and bonding ability similar to GIC's while maintaining the high aesthetic qualities of composite resins (Salama & El-Mallakh, 1997). Dyract AP^a introduced by Dentsply some years ago is one

such compomer that combines the properties of composites and glass-ionomer cements (Dyract AP Technical Manual, 1997).

The setting reaction of compomers differs from that of glass-ionomer cements and composites. Composite setting reaction consists of joining monomer molecules into one cross-linked network. Uncured composites consist essentially of a mixture of resinous monomer and an inert glass powder. Since the glass is inert, it is normally coated with a reactive, polymerisable layer to enable it to become chemically part of the cured composite. Curing the composite then simply joins the monomer molecules and therefore a set composite consists of an inert glass in a matrix of organic polymer. On the other hand compomers setting reaction occurs through light activation of the resin matrix as in a composite (Dyract AP Technical Manual, 1997). An acid base reaction between the strontium fluorosilicate glass and the carboxylic groups occurs in the presence of water in the oral cavity which leads to further cross-linking of the polymer and the release of fluoride (Salama & El-Mallakh, 1997).

Light cured polyacid-modified composites stored in distilled water will undergo a slow-rate, solid state transformation to produce carboxylate salts and reach a saturation point after 4 weeks. The carboxylate salt-rich layer measures about 100 μ m deep from the surface and is formed after 4 weeks of storage in deionized water. It is expected that initially carboxylate salts are formed with the divalent ions strontium and barium and

later a polysalt matrix is established by aluminum-carboxylate complexes (Eliades *et al.*, 1998).

The rate and extent of the acid-base reaction in polyacid-modified composites are dependent on many factors which may substantially modify the clinical outcome. Ionization of carboxyls from interfacial water may promote adhesion to dental tissues. However, the water deficiency at these regions compared with the free surface and the presence of bonding systems may substantially modify the ionization potential and consequently the salt formation yield with enamel and dentine. In low pH aqueous environments, polyacid-modified composites demonstrate increased solubility and selective dissolution of barium, aluminum, strontium and calcium, by forming water-soluble salts which may further retard or completely inhibit the carboxylate salt yields on the material surfaces. Another factor that may influence the reaction is the curing light intensity. Higher light intensities would improve the polymer network formation but may reduce the mobility of the carboxyl groups attached to the monomer backbone and may subsequently prolong the time required to obtain a salt yield (Eliades *et al.*, 1998).

However, compomers do not set as a consequence of an acid-base reaction and the acid-base reaction does not occur until after photo-polymerization and diffusion of water in the set material. Thus, although compomers contain ingredients of glass-ionomers they are in insufficient amounts to promote acid-base curing, and therefore cannot be classified as glass-ionomer cements (McLean *et al.*, 1994). Compomers fall into a

category of their own with glass-ionomer cement at one end and composite resin cement at the other end of the dental material spectrum with compomers lying somewhere between these two. Thus the development of polyacid-modified resin composites effectively completes the transition from materials with a water-based setting reaction to those that are almost entirely resin based (Watson, 1999).

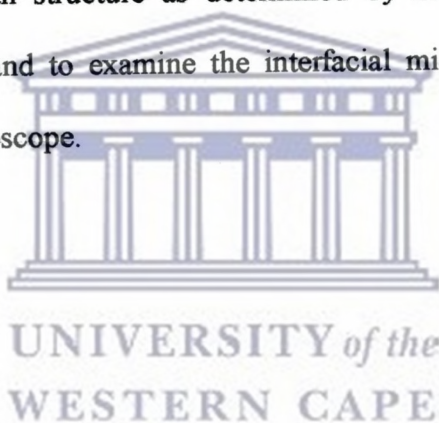
Meyer *et al.* (1998), in an *in-vitro* comparative study concluded that compomers behave more like composite resins than glass-ionomers. The lack of setting in the absence of light, the small amount of sorbed water, the consequently lower effect of water on the material's stiffness, the higher values of the mechanical properties and a severely reduced release of fluoride are signs of a closer relationship to composites than to glass-ionomers. When the compomer Dyract was compared to a resin-modified glass ionomer cement Fuji II LC, the two materials behaved differently when stored in the presence of water. In contrast to Fuji II LC, Dyract showed very little expansion for the first 24 hr, leached small quantities of ions and retained its mechanical strength (Cattani *et al.*, 1999). A comparative shear bond strength study of a compomer Dyract (10.27 MPa) to that of a composite Herculite with Optibond (17.61 MPa) showed that the composite did exhibit a significantly higher shear bond strength (Jumlongras & White, 1997).

Adhesion of polyacid-modified composites to tooth structure should theoretically be via a dual mechanism. The resin portion of the cement offering a micromechanical bond through hybrid layer and resin tag formation and the glass-ionomer component presenting

a diffusion-based adhesion to the tooth structure. Despite some evidence of glass-ionomer type setting reactions, there is no inherent adhesion of polyacid-modified composites to tissue. It is essential, therefore, that an intermediary adhesive is applied to the tooth. Many of the adhesives that have been produced for use with polyacid-modified composites are of the self-etching variety. Manufacturers claim that etching with phosphoric acid is not necessary. The low pH of the adhesive is sufficient to remove the smear layer and produce a resin infiltration zone in dentine. However, penetration was found to be greatly reduced when compared with the same adhesive applied following phosphoric acid etching of the dentine. The penetration of resin tags into the enamel is also greatly reduced. There is therefore a risk of failure in both dentine and enamel at these interfaces (Watson, 1999). When the microleakage of Dyract AP on etched and unetched tooth surfaces was compared, Kugel *et al.* (1998) found that the compomer applied to etched tooth surface demonstrated less microleakage than when applied to unetched tooth surface. When Dyract was used with Prime&Bond 2.0 the length of the resin tags were about 100 μ m and when Dyract was used with Dyract-PSA primer the resin tags ranged from 1-6 μ m in length. Also, the best dentinal seal was obtained when Prime&Bond 2.0 was used (Chersoni *et al.*, 1997).

Because many of the newer adhesives that have been produced for use with polyacid-modified composites are of the self etching variety, it is important to compare the adhesive properties of these self etching adhesives with that of the traditional three component etching, priming and bonding type of adhesives. Further, studying the

interfacial relationship between restoration and tooth structure using a confocal scanning laser microscope provides an advantage in that the distribution of the both types of the adhesive systems can be studied and compared *in-situ* thereby giving a better understanding of its adhesive properties to tooth structure. Also, very few reports are available in the literature on the physical properties and clinical evaluation of polyacid-modified composite resins as these materials are fairly recent. Therefore, the aim of this research is to study one of the physical properties of polyacid-modified composites, namely its adhesion to tooth structure as determined by shear bond strength and microleakage tests *in-vitro* and to examine the interfacial micromorphology using a confocal scanning laser microscope.



CHAPTER III

Materials and Methods

In this laboratory study two compomers were evaluated with two adhesive systems, namely:

- a). Dyract AP^a with Non-Rinse Conditioner (NRC)^a and Prime&Bond NT^a
- b). F2000^b compomer with Scotchbond Multi-Purpose Plus^b adhesive (table 2).

A total of 60 extracted non-carious human premolar and molar teeth were used *i.e.* 30 teeth for each compomer system of which 15 molars were randomly selected and used for shear bond strength determinations and 15 premolars for microleakage determinations. The teeth were stored in physiological saline solution containing a few crystals of thymol (to prevent mold formation) at room temperature for less than a month until used. All teeth were carefully cleaned with flour of pumice and screened under a dissecting microscope^f at x10 magnification for any damage prior to cavity preparation and only undamaged teeth were used.

Material	Composition	Lot no.	Manufacturer
Dyract AP	Polymerizable Resins TCB Resins Strontium-fluoro-silicate glass Strontium fluoride Photoinitiators	98006000670 Expiry date: 2000 02	Dentsply, DeTrey Addlestone Surrey U.K.
NRC	Organic acids/ monomers	9806000046 Expiry date: 2001 04	Dentsply, DeTrey Addlestone Surrey U.K.
Prime &Bond NT	Methacrylate resins Amorphous silica PENTA Photoinitiators Cetylamine hydrofluoride Acetone	9810000584 Expiry date: 2000 09	Dentsply, DeTrey Addlestone Surrey U.K.
F2000	FAS glass-fluoride CDMA oligomer (dimethacrylate oligomer from citric acid) GDMA (glyceryl dimethacrylate) Hydrophylic polymer	19980714 Expiry date: 2000 05	3M Dental St. Paul, Minn., USA
ScotchBond MP	Primer: HEMA Polyalkenoic acid copolymer Adhesive: BIS-GMA HEMA resin Amines	19980105 Expiry date: 2000 12	3M Dental St. Paul, Minn., USA

Table 2. Restorative systems used (composition according to manufacturer).

Shear bond strength tests

Thirty extracted non-carious human molars (15 for each test group) were used for shear bond strength determination. The teeth were cleaned with flour of pumice on a dental lathe and stored in physiological saline containing a few crystals of thymol at room temperature for less than 2 months. The roots of the teeth were removed with a separating disc and the bulk of the occlusal enamel removed by grinding wet using a 60-grit silicon carbide paper^c on a polishing machine^d. The teeth were embedded in PVC containers^e with cold cure acrylic resin so that the ground occlusal surfaces projected well above the embedding medium (figure 12).

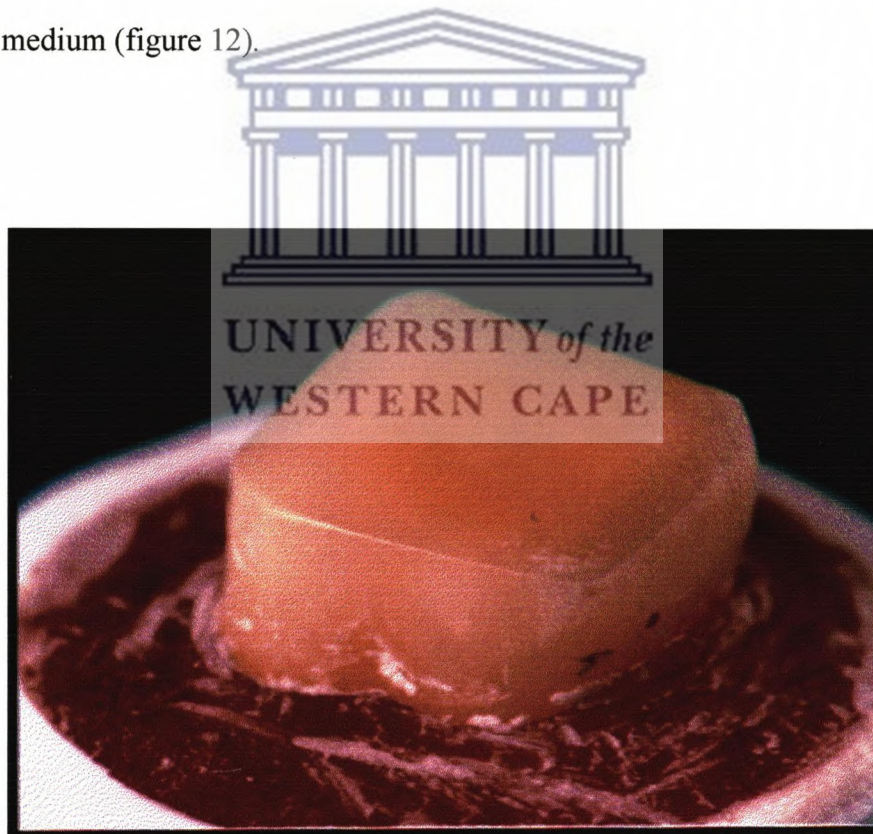


Figure 12. Tooth embedded in PVC container with resin.

The PVC containers were prepared by cutting 10 mm lengths from a long PVC pipe (with an outer diameter of 20 mm and a wall thickness of 2 mm). Immediately after embedding, the superficial dentine was exposed by grinding wet on 180-grit followed by 600-grit silicon carbide paper^c on a polishing machine^d. The ground dentine was examined under a dissecting microscope^f at x50 magnification to ensure that no remnants of enamel remained on the ground surface. The dentinal surfaces were washed thoroughly under a stream of tap water and air dried for ten seconds. A Wilkerson air filter^g is attached to the laboratory air line which effectively removes all contaminants. Annular discs with an inner diameter of 3mm were punched into adhesive tape no. 471^b and the perforated adhesive tape stuck onto the ground dentine surfaces to demarcate the bonding areas. The margins of the tape were burnished to obtain a good seal between the tape and the dentine. The embedded teeth were randomly divided into two groups of 15 teeth each. Group A was used for shear bond strength testing for Dyract AP with Non-Rinse Conditioner (NRC) and Prime&Bond NT (PBNT) while Group B was used for the second compomer, namely F2000 with Scotchbond Multi-Purpose Plus (SBMP) dental adhesive system.

In Group A, NRC was applied to the dentine in the demarcated area using an applicator tip^a according to the manufacturer's instructions (figure 13). This was left undisturbed for 20 seconds and excess was removed with an air syringe taking care not to desiccate the dentine.

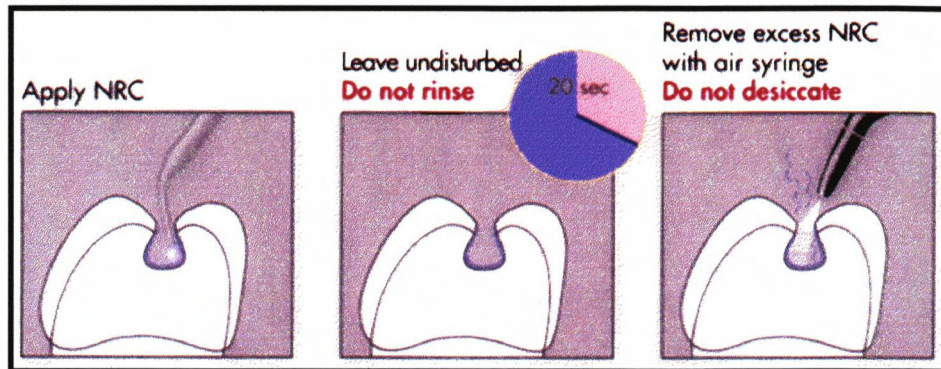


Figure 13. Manufacturer's instructions for the application of NRC.

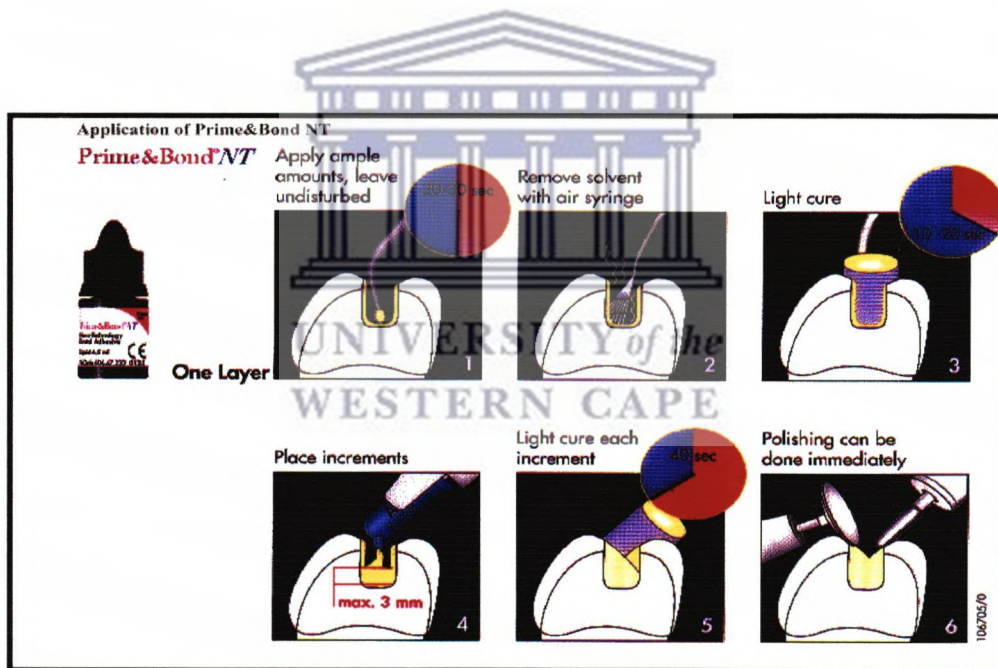
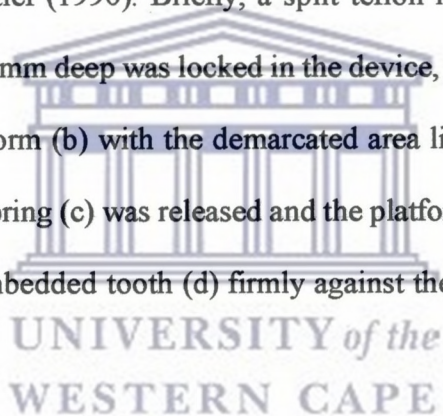


Figure 14. Manufacturer's instructions for the application of PBNT.

Prime&Bond NT was then applied in ample amounts to the surface according to the manufacturer's instructions (figure 14). This was left undisturbed for 20 seconds and then air syringed to remove excess solvent. The adhesive was light cured for 20 seconds using an Optilux visible light curing unit^h. This unit was monitored with a light meterⁱ prior to use and the light output was 350mW/cm².

The teeth were then mounted in the assembly apparatus (figure 15) as described extensively by Kamel & Retief (1990). Briefly, a split teflon mold (a) with a circular hole, 3 mm in diameter and 5 mm deep was locked in the device, and the embedded tooth compressed against the platform (b) with the demarcated area lined up with the hole in the teflon mold. The finger spring (c) was released and the platform elevated to compress the dentine surface of the embedded tooth (d) firmly against the base of the split teflon mold.



A small increment of Dyract compomer was transferred to the opening in the teflon mold, compressed and light cured for 40 seconds. The filling was done in three increments. Fifteen minutes after final cures the test specimens were disassembled and stored in physiological saline at 37°C.

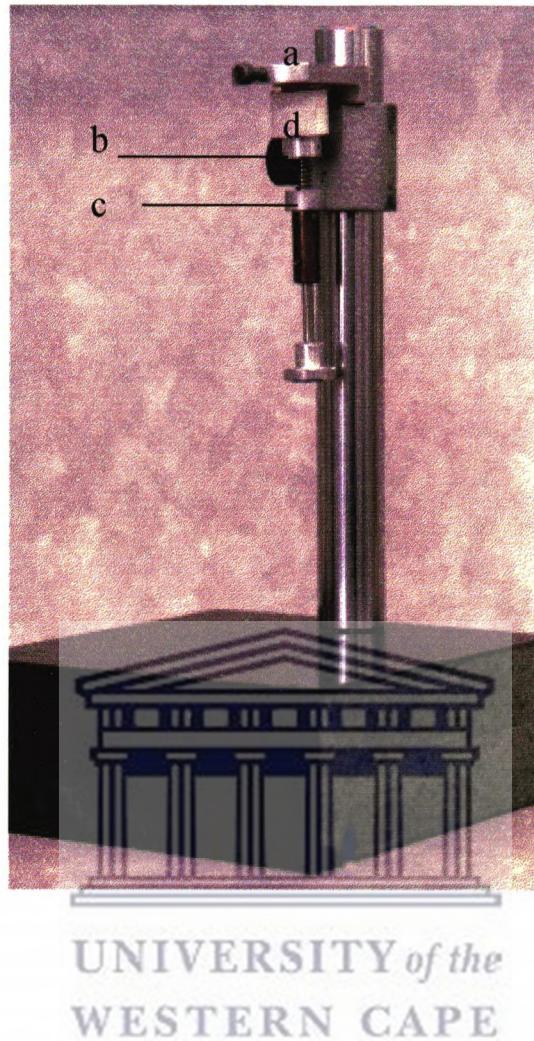


Figure 15. Assembly apparatus. a. Teflon mold
b. Platform
c. Fingerspring
d. Embedded tooth in PVC container.

In Group B, F2000 compomer system was used with Scotchbond Multi-Purpose Plus (SBMP) to test the shear bond strength. The bonding agent was applied according to manufacturer's instructions (figure 16).

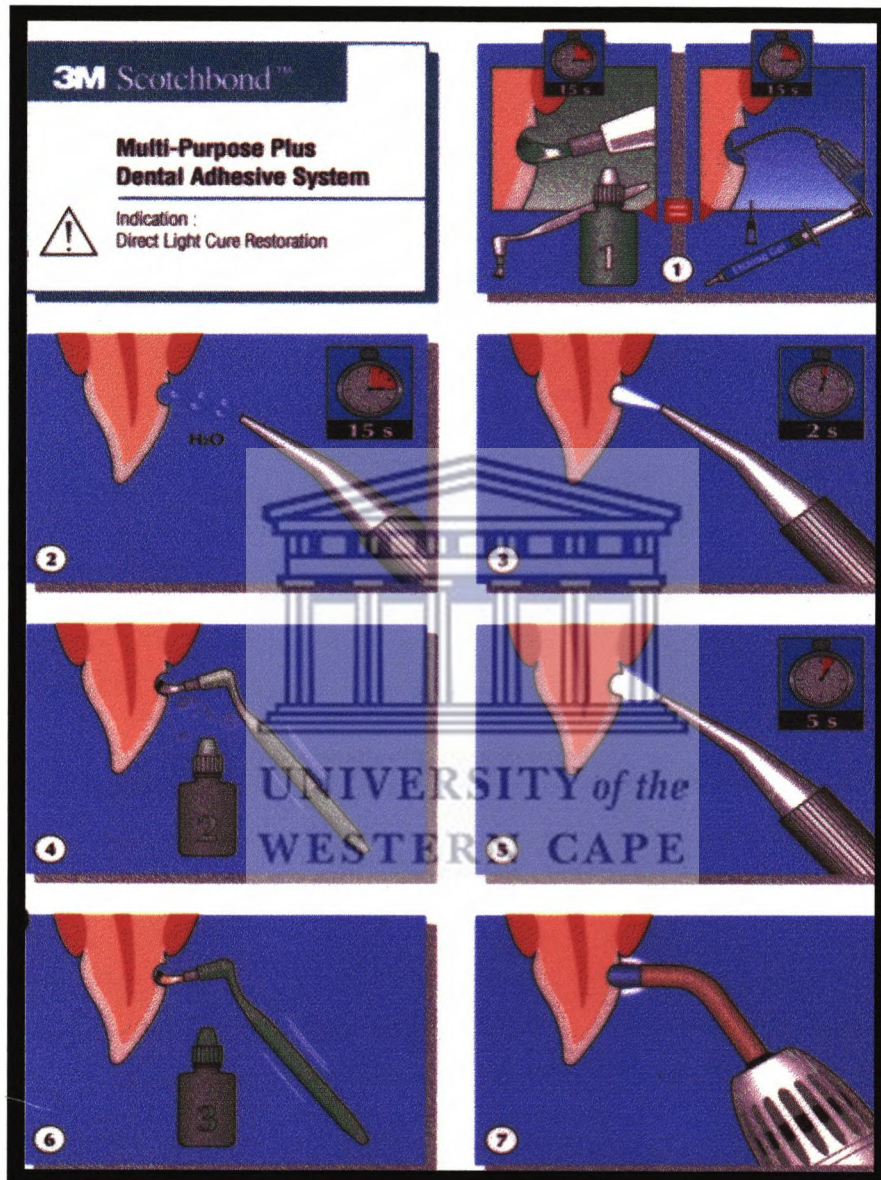


Figure 16. Manufacturer's instructions for the application of SBMP

Briefly, the dentine in the demarcated area of the perforated adhesive tape was etched

with 37% phosphoric acid for 15 seconds. The etched dentine was rinsed thoroughly with water for 15 seconds and air dried for 5 seconds taking care not to desiccate the dentine. Scotchbond Multi-Purpose (SBMP) primer was applied to the etched surface, left undisturbed for 5 seconds and then air-dried. Following this SBMP adhesive was applied and light cured for 10 seconds using Optilux visible light curing unit^h. The teeth were then transferred to the assembly apparatus containing the teflon mold as described above and filled with F2000 (figure 17).

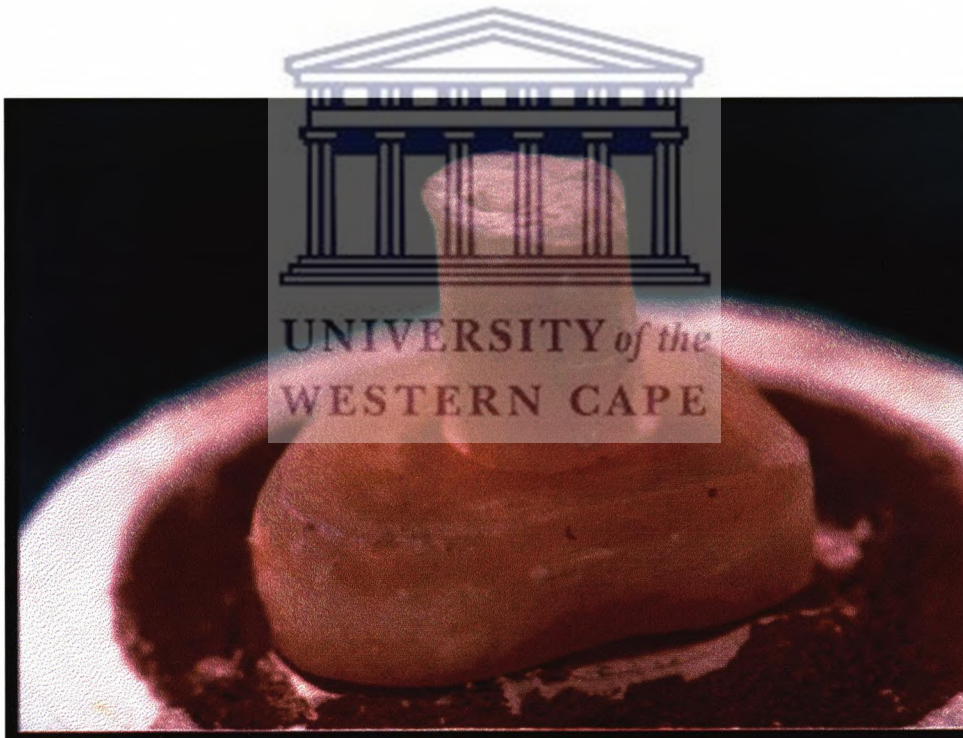
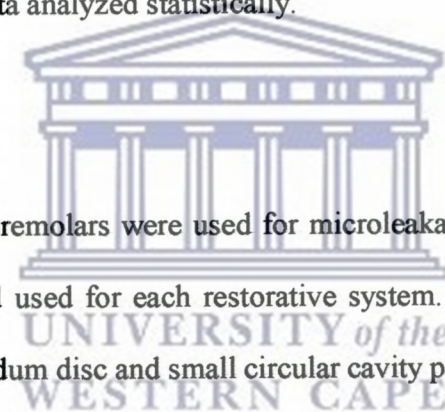


Figure 17. Tooth with restoration embedded in PVC container

This was done in three increments and light cured at 40 seconds per increment. The samples were then stored in physiological saline at 37° C. For shear bond strength testing the Zwick universal testing machine^j was used. The specimens were locked in a special device which was clamped to the base of the Zwick machine. A shear load was applied vertically to the base of the bonded compomer cylinder by means of a knife-edged rod. The crosshead speed was 0.5 mm/minute. The internal calibration of the testing machine was correlated with external weights. The shear bond strengths were calculated and expressed in MPa and the data analyzed statistically.

Microleakage tests

Thirty non-carious human premolars were used for microleakage studies. Fifteen teeth were randomly selected and used for each restorative system. The apices of the roots were cut off with a carborundum disc and small circular cavity preparations were made at the cut surfaces with an inverted cone carbide bur (SS White FG-35^k). Two coats of Copalite varnish^l were applied to the preparations and restored with amalgam. This procedure effectively eliminates microleakage at the root apices (Retief *et al.*, 1994). Cylindrical cavities were prepared on the facial surfaces of the premolars. Cavities 3mm in diameter and 3mm deep were cut with a cavosurface angle of 90° using a carbide fissure bur (SS White FG-331^k). The burs were replaced after 6 cavity preparations. To reproduce conditions approximating those in normal clinical practice, the cavities were prepared using a high speed handpiece with water coolant.



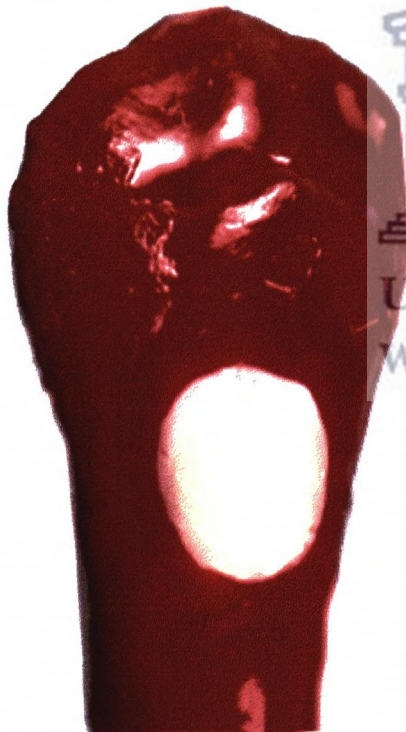
For the microleakage studies of the compomer Dyract AP with NRC and Prime&Bond NT, the steps included were detailed by the manufacturer (figure 13 & 14). Briefly the steps followed were:

After cavity preparation the surface was washed thoroughly with air/water spray. The water was then removed by gentle blow drying without desiccating the preparation.

1. Sufficient amounts of NRC (Non Rinse-Conditioner) was applied with an applicator tip^a and left undisturbed for twenty seconds. Excess NRC was removed by gentle blow drying with the air syringe.
2. PBNT was applied with an applicator tip to the cavity preparation to thoroughly wet the cavity surface and left undisturbed for 20 seconds. Excess solvent was removed with an air spray for 3-5 seconds. Using a visible curing light^h the Prime&Bond NT was cured for 20 seconds.
3. Dyract AP was inserted into the cavity preparation in increments using an applicator gun with compules.
4. Each increment was cured for 40 seconds using a visible curing light^h.
5. The restoration was finished immediately after curing. A series of graded Sof-Lex^b discs were used for final finishing and polishing of the restoration.

For the microleakage studies of the compomer F2000 and Scotchbond Multi-Purpose Plus, the steps followed were:

1. The cavity preparations were etched with 37% phosphoric acid for fifteen seconds and thoroughly rinsed for fifteen seconds. It was then air dried for five seconds leaving the surface moist. SBMP primer was then applied to the etched enamel and dentine and gently air dried for five seconds.
2. SBMP adhesive was applied to the primed enamel and dentine and light cured for 10 seconds.
3. The cavity was restored with F2000 compomer in increments and each increment light cured for 40 seconds.
4. Final finishing and polishing was achieved with a series of polishing disks (Sof-Lex^b).



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Teeth from both samples were coated with two layers of nail varnish^m leaving a 1 mm window around the restoration free of varnish (figure18).

Figure 18. Tooth coated with varnish leaving a 1mm window around the restoration.

All 30 teeth were then cycled in 2% basic fuschin solution for 500 cycles in a thermocycling machine with temperatures ranging from 15° C to 45° C and a dwell time of 15 seconds. The teeth were removed from the thermocycling machine and the nail varnish cleaned off with flour of pumice. The samples were embedded in resin and sectioned with a diamond disk cutter (Struers Minitomⁿ) using a 350 μ m blade thickness. Tooth sections of 400 μ m were cut in running water at a speed of 250 rpm.

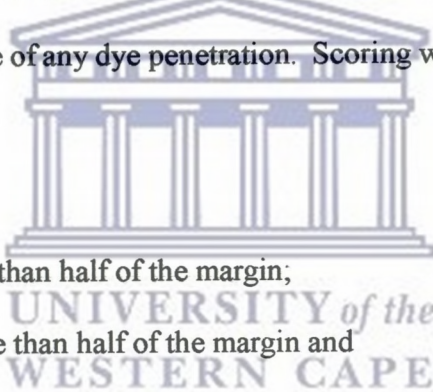
The microleakage of the specimens were then evaluated under a light microscope^f at x60 magnification for the presence of any dye penetration. Scoring was done according to the following criteria:

0 = no penetration of dye;

1 = penetration of dye to less than half of the margin;

2 = penetration of dye to more than half of the margin and

3 = penetration of dye to the axial wall (Puckett *et al.*, 1995).



Confocal scanning laser microscopic study

Three non-carious human molars were used for each material and treated as outlined for the shear bond strength determination. However, a fluorescent dye was incorporated in the adhesive resin of each compomer system to be able to visualize the distribution of resin within the dentine. Rhodamine B isothiocyanate^o fluorescent dye was used at a concentration of 0.066%. This was dissolved easily and homogeneously into the primer of Scotchbond Multi-Purpose Plus and NRC. Fluorescein^o dye, also at a 0.066% concentration was incorporated into the adhesive of Scotchbond Multi-Purpose Plus and into the Prime&Bond NT.

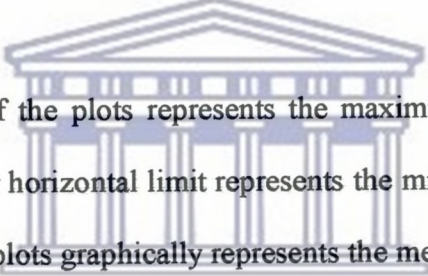
The teeth were then sectioned longitudinally using a diamond disk cutterⁿ. The sections were polished up to 1000- grit fineness. The confocal microscope of the type Zeiss LSM 410^P was used for examination of the cut sections. The CSLM is a variable-energy mixed-gas laser using a krypton/argon-mixed gas laser. Samples were selected randomly and examined to ascertain the fluorescence distribution, hybrid layer, if visible and the presence or absence of resin tags. Optically, layers were viewed below the layer that was disturbed by cutting or sectioning. Initially, the sections were examined under lower magnification (x20) to give an overall picture of the adhesive distribution. Samples were then examined under x60 oil immersion objectives for details of the interface and areas such as cavity lines. The fields of view were digitally recorded on CD-Rom using LSM software available with the Zeiss confocal microscope unit.

CHAPTER IV

Results

Shear bond strength tests

The Violin Plots (figure 19) graphically display the distribution of the shear bond strength values obtained for Prime&Bond NT (PBNT) and ScotchBond MP (SBMP). The median, quartiles, maximum and minimum values are depicted.



The upper horizontal limit of the plots represents the maximum shear bond strength values obtained and the lower horizontal limit represents the minimum values obtained. The yellow dot on the violin plots graphically represents the median values obtained for the respective material while the green line extending from the yellow dot depicts the interquartile range representing the middle 50% of the values. The thin red line extending above the green line represents the upper 25% of the shear bond strength values obtained while the red line below represents the lower 25% for each material.

Violin Plots are particularly useful for comparing groups of observations. The visual description of data provides an overview of the range and the distribution of the individual values obtained.

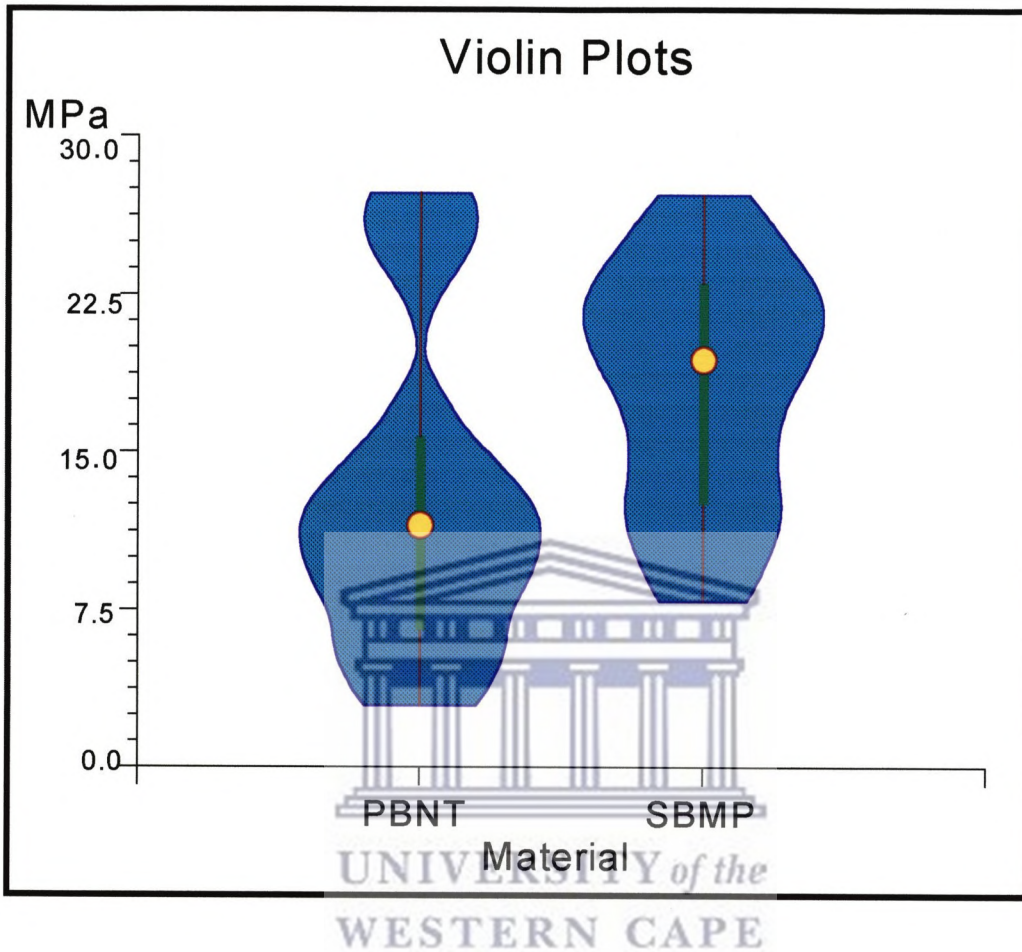


Figure 19. Violin Plots comparing shear bond strength of PBNT and SBMP to dentine.

When the data were subjected to a non-parametric statistical analysis, there was a significant difference between the two groups ($p < 0.05$). In this study the Mann-Whitney U test was used and the results show that the shear bond strength of SBMP to dentine was significantly higher than the shear bond strength of PBNT to dentine ($p < 0.05$).

Microleakage tests

The comparative microleakage scores for SBMP and PBNT on enamel (occlusal) and dentine (gingival) portions of the restorations are shown in tables 3 & 4.

Leakage scores on enamel margin

<i>Material</i>	<i>Score 0</i>	<i>Score 1</i>	<i>Score 2</i>	<i>Score 3</i>
PBNT	13	2	0	0
SBMP	15	0	0	0

Table 3. Comparative microleakage scores for PBNT and SBMP on enamel margin

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Leakage scores on dentine margin

<i>Material</i>	<i>Score 0</i>	<i>Score 1</i>	<i>Score 2</i>	<i>Score 3</i>
PBNT	2	13	0	0
SBMP	15	0	0	0

Table 4. Comparative microleakage scores for PBNT and SBMP on dentine margin

The microleakage values were subject to Fisher's Exact Test after collapsing scores 1-3. On the dentine side of the restorations the statistical results show that there was a significant difference between the two materials ($p < 0.0001$). It was found that PBNT showed more leakage than SBMP on the dentine side. In this study SBMP showed no microleakage on both the enamel and dentine sides.

For both the materials used, there was no significant difference between the microleakage values on the enamel side of the restorations ($p > 0.05$). PBNT showed significantly greater microleakage on the dentine side than the enamel side ($p < 0.05$).

Confocal scanning laser microscopic study

Images of the dentine/restorative interface of SBMP and PBNT are shown in figures 20, 21, 22, & 23. The visualization and distribution of the bonding agents were clearly seen with the use of CSLM. This was greatly enhanced by the incorporation of fluorescent labels. Rhodamine B gave a bright orange red fluorescence when excited with green light of 568nm wavelength and 575-640nm wavelength band-pass filter. Fluorescein was detected when an excitation filter of 488nm and a band-pass filter of 500-530nm were incorporated into the system.

Figure 20 shows fluorescence only visible from the rhodamine B and represents the interface between dentine (D) and NRC primer labelled with rhodamine B. The penetration of NRC into the tubules can be clearly seen with the formation of resin tags (RT). Funneling at the opening of the tubules is evident indicating opening of the dentinal tubules and penetration of the NRC. Length of resin tags was found to be about $10\mu\text{m}$. A distinct hybrid layer (H) is formed at the interface between the adhesive layer (A) and the dentine (D). Hybrid layer thickness is about $2\mu\text{m}$. Adhesive layer (A) is mainly the combination of NRC and PBNT adhesive resin with NRC expected at the base. (x2800 / excitation filter 568nm; band-pass filter 575/640nm).

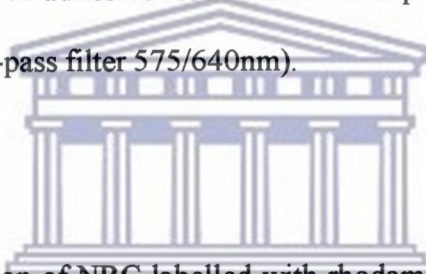


Figure 21 shows a combination of NRC labelled with rhodamine B and PBNT labelled with fluorescein. The bottom part of the yellow band (A) represents the combination of the rhodamine in NRC and PBNT. The rest of the yellow band contains mostly PBNT while the green/yellow portion representing the hybrid layer (H). Resin tags (RT) can be clearly seen indicating penetration of the PBNT into the dentine (D). The length of the resin tags were found to be about $10\mu\text{m}$. (x850 / excitation filter 488nm; band-pass filter 500/530nm).

The adhesive in SBMP (A) labelled with fluorescein, penetrates deep into the dentine as seen in figure 22. Resin tags (RT) clearly follows the path of the dentinal tubules into the dentine (D). F2000 compomer (R) was used as the restorative material. (x280/ excitation filter 488nm; band-pass filter 500/530nm).

Figure 23 shows a combination of SBMP primer labelled with rhodamine B and the SBMP adhesive labelled with fluorescein. Long resin tags (RT) are formed in the dentine (D) and extending close to the pulp (P). The resin tags measure about 150 μ m long. The hybrid layer (H) can be seen at the interface between the adhesive layer and the dentine. This measured about 5 μ m in thickness. The red/yellow portion shows a mixture of adhesive and primer. F2000 compomer (R) was used as the restorative material. (x350/ excitation filter 568nm; band-pass filter 575/640nm).



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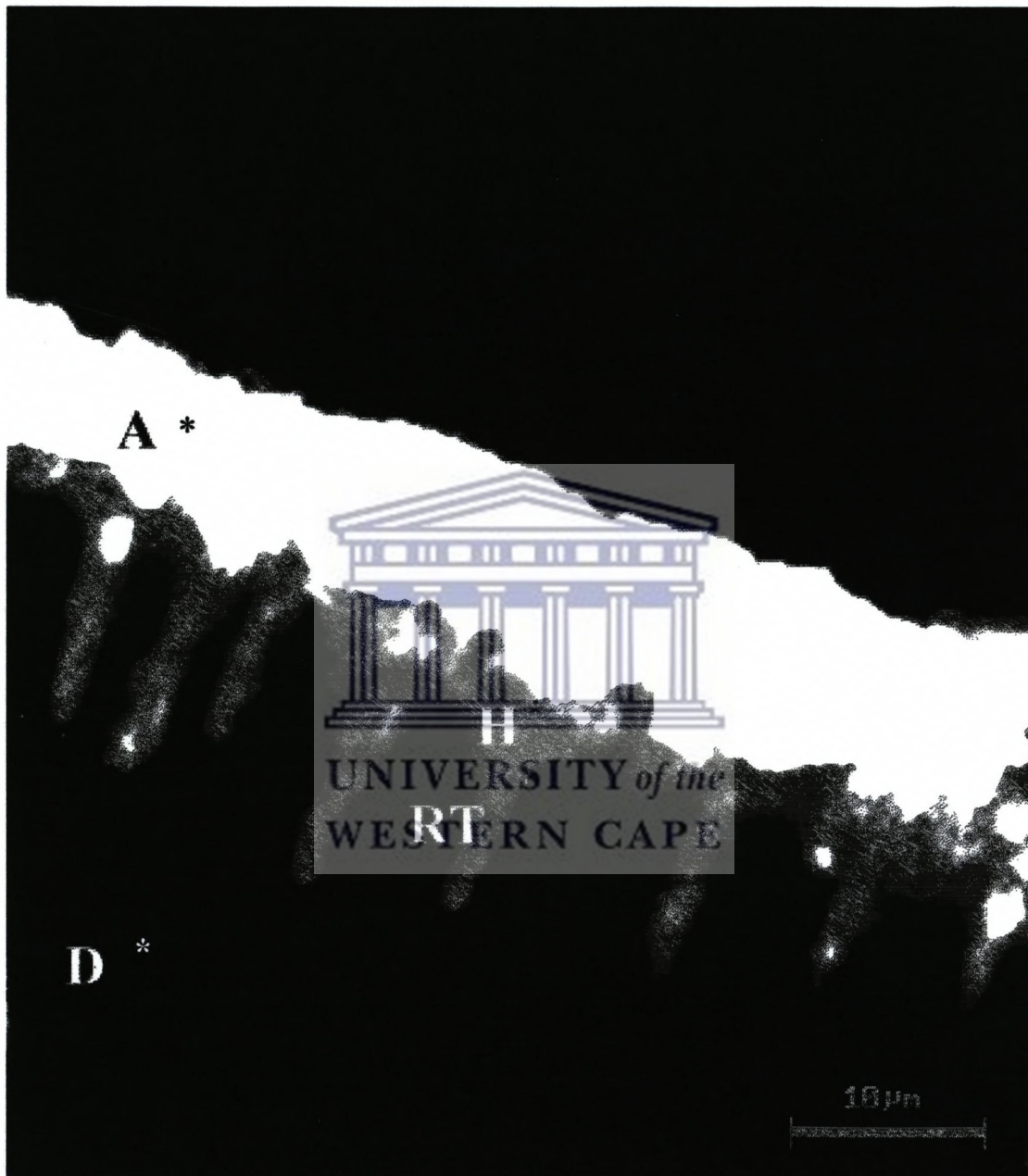


Figure 20. Interface between dentine (D) and adhesive layer (A). NRC labelled with rhodamine B showing resin tags (RT) in the dentine. The hybrid layer (H) can be seen at the interface. x2800 / excitation filter 568nm; band-pass filter 575/640nm.

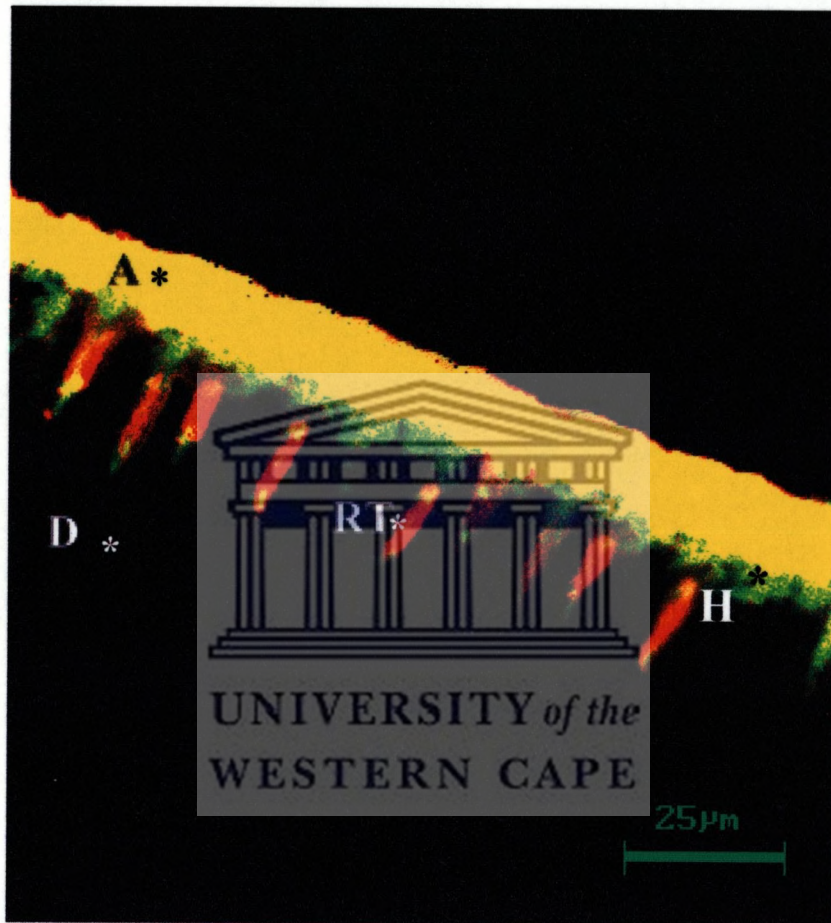


Figure 21. Interface between dentine (D) and adhesive layer (A) which consists mainly of PBNT. The hybrid layer is represented by the greenish band (H). NRC labelled with rhodamine B and PBNT labelled with fluorescein showing resin tags (RT) extending into the dentine. x850 / excitation filter 488nm; band-pass filter 500/530 nm.

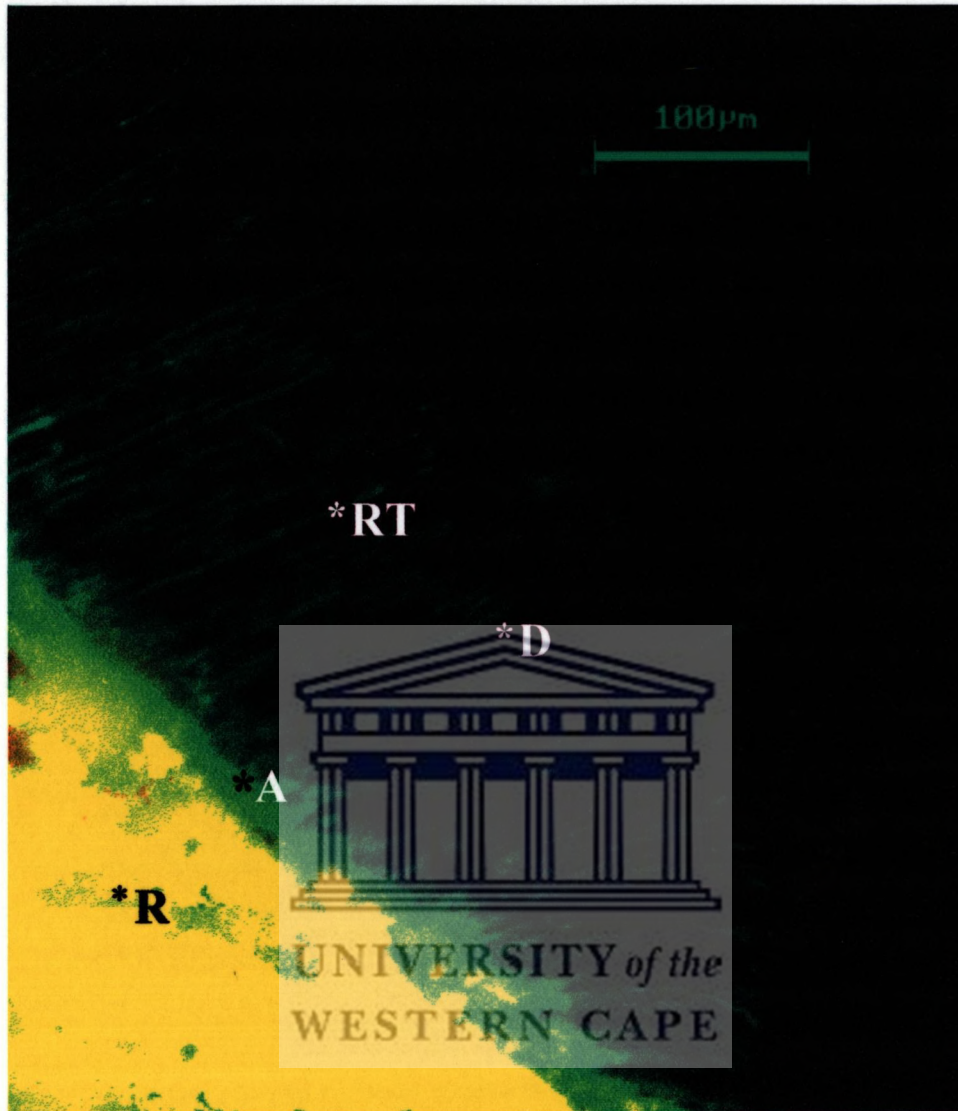


Figure 22. Interface between dentine (D) and SBMP adhesive (A). SBMP labelled with fluorescein showing resin tags (RT) extending into the dentine. The tooth was restored with F2000 compomer(R). x280 / excitation filter 488nm; band-pass filter 500/530nm.

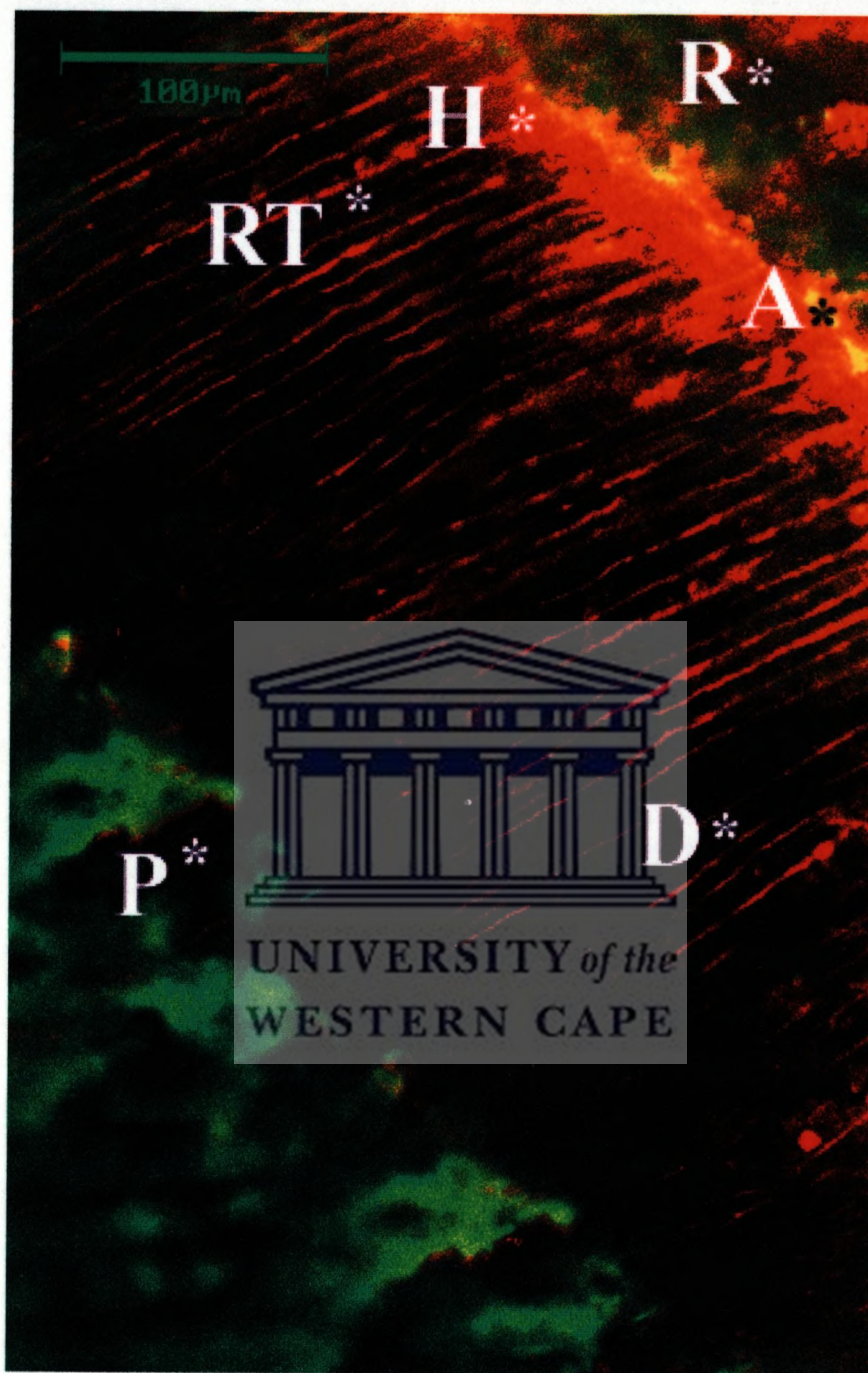


Figure 23. Interface between dentine (D) and SBMP primer labelled with rhodamine B and SBMP adhesive labelled with fluorescein showing hybrid layer (H) and resin tags (RT) extending close to the pulp (P). The red-yellowish band (A) represents a mixture of the adhesive and primer. The tooth was restored with F2000 compomer(R). x350 / excitation filter 488nm; band-pass filter 500/530nm.

CHAPTER V

Discussion

Mechanical testing of adhesion provides some knowledge of the adhesive properties of dental restorative materials. Shear bond strength and quantitative microleakage are strongly associated with each other both due to statistical and physical/mechanical evidence (Retief *et al.*, 1994). It has been suggested that dentine bonding agents should have a shear bond strength of at least 17 MPa to eliminate microleakage (Jumlongras & White, 1997). Thus a material with a strong initial bond to tooth structure is needed to resist polymerization shrinkage and microleakage.

Requirements for an effective dentine adhesive system include the ability to thoroughly infiltrate the collagen and partially demineralized zone, to co-mingle and encapsulate the collagen and hydroxyapatite crystallites to produce a well polymerized hybrid layer (Nakabayashi *et al.*, 1992). Optimal bond strength is derived from complete resin diffusion into the chemically altered dentine. Failure to adequately penetrate the collagen network into the partially demineralized dentine may produce a weak porous layer of collagen not protected by hydroxyapatite or encapsulated by resin. Subsequent hydrolysis of the exposed collagen could lead to degradation of the bond, resulting in decreased bond strengths and increased microleakage over time (Sano *et al.*, 1995).

Removal of the organic collagen layer following acid conditioning and subsequent bonding directly to the partially demineralized hydroxyapatite component of the dentine layer may produce more durable adhesion in some adhesive systems and not in others (Vargas *et al.*, 1997a). Deeper penetration of resin into the tubules and anastomosis was observed when this collagen was removed. Removal of the collagen layer with NaOCl enhanced the bond strength of All-Bond 2 but not Scotchbond Multi-Purpose. In that study the shear bond strength of Scotchbond Multi-Purpose was found to be 17.37 MPa before removal of the collagen layer and there was no significant difference after removal of this layer. Vargas *et al.* (1997a) suggests that this may be due to the efficient penetration of SBMP primer as was found in this study.

CSLM images in this study show SBMP primer penetrating into the demineralized dentine and deep into the dentine tubules to form long resin tags (figures 22 & 23). Several factors may affect the ability of the primer to adequately penetrate into the demineralized dentine. The acid conditioning used with SBMP (37% phosphoric acid for 15 seconds) demineralizes the dentine and opens the tubules. Rinsing with water removes the smear layer and smear plugs thereby allowing proper penetration of the primer. Scotchbond Multi-Purpose primer consists of HEMA (hydroxyethyl methacrylate) and a polyalkenoic acid copolymer. HEMA is a hydrophilic acidic primer capable of penetrating the tubules and thereby allowing the subsequent layer of adhesive to “wet” the etched surface. The subsequent application of the adhesive resin, which consists of

Bis-GMA and HEMA, then penetrates deep into the dentine tubules and demineralized dentine forming long resin tags and a distinct hybrid layer as seen in the CSLM images.

The results of this study disclosed no microleakage for the Scotchbond MP. Good seals were obtained on both enamel and dentine sides. This suggests good penetration of the adhesive into the demineralized enamel and dentine.

The shear bond strength of SBMP in this study was 18.1 MPa and this compares favourably to other researchers (figure 24).

Unlike the SBMP system that uses a separate acid-etch technique of 37% phosphoric acid, the NRC system uses 10% maleic acid to condition the dentine surface in combination with a primer using a non-rinse technique. Because this is a non-rinse technique the smear layer is not rinsed off. The smear layer according to the manufacturer is incorporated into the adhesive layer. However, the presence of the smear layer remnants could inhibit the penetration of the primer. The NRC is composed of organic acids and acidic monomers dissolved in water. The conditioning acid (maleic acid) contained in the NRC is used to cleanse and condition the tooth surface (table 5).

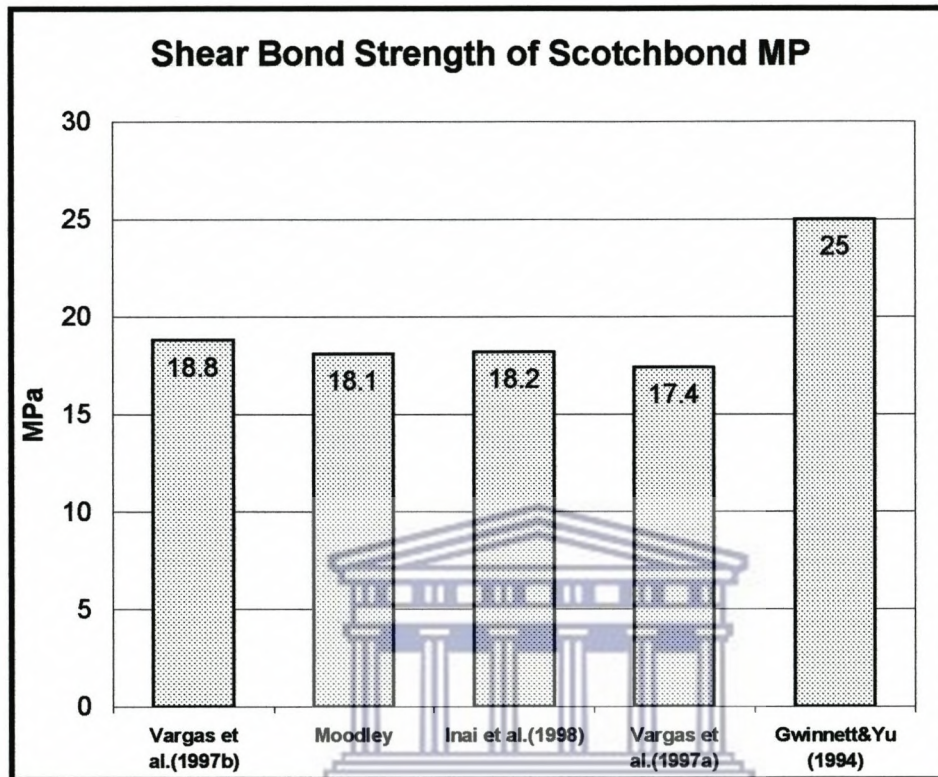


Figure 24. Comparative shear bond strengths of SBMP to dentine.

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The NRC also contains an organic priming acid (itaconic acid). This acid contains methacrylate-type double bonds that can polymerize with the adhesive resin. Furthermore, the priming acid contains carboxylic acid groups that are capable of adhering to the calcium ions of the tooth structure (NRC Technical Manual, 1998).

Dentine is composed of hydroxyapatite and organic material, mainly collagen. During acid etching the calcium ions are removed from the dentine, and the possibility for

formation of calcium bridges between the carboxylic group and dentine is greatly reduced (Ruyter, 1992). Furthermore there is an enrichment of collagen at the surface of dentine during acid etching, so the bonding to dentine is weakened due to the decreased bridge formation by calcium ions and reduced quantities of apatite at the dentine surface. Better adhesion via calcium ion bonding to the enamel than to dentine may be present and this may explain the higher microleakage found on the dentine side rather than on the enamel side in this study. Dyract AP with NRC and Prime&Bond NT showed no significant microleakage on the enamel side but showed significantly greater microleakage on the dentine side when compared to leakage on the enamel side.

Component	Function
Conditioning acid (maleic acid)	Conditions and cleanses the tooth
Priming acid (itaconic acid)	Primes the tooth surface Copolymerizes with Prime&Bond NT; It's carboxylic group adheres to calcium of the tooth
Water	Solvent

Table 5. Composition of NRC and their functions (NRC Technical Manual, 1998).

The mean shear bond strength obtained for NRC plus Prime&Bond NT to dentine was 12.8 MPa in this study. This result was compared to that of other authors (figure 25). Values for the respective authors were obtained from NRC Technical Manual (1998).

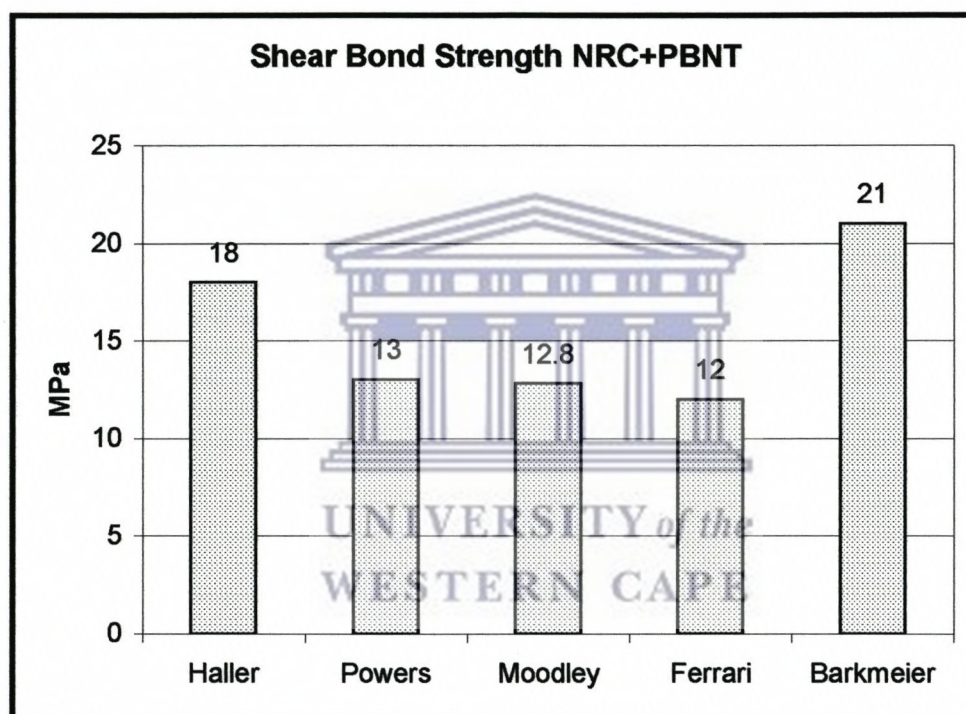


Figure 25. Comparative shear bond strengths of NRC & PBNT to dentine.

According to the manufacturer, NRC was designed specifically to improve adhesive properties of the tooth surfaces.

Van Meerbeek's report to Dentsply DeTrey (NRC Technical Manual, 1998) states:

- "... the application of NRC completely removed the smear layer, exposing a microporous collagen scaffold. The dentine tubules were completely deprived from the smear layer plugs and a circularly oriented collagen fibril network was exposed at the tubule orifice walls".
- "...intertubular dentine was demineralised for approximately 1-2 μm . The typical shag carpet appearance with upwards directed collagen fibrils can easily be recognised. Areas of this typical shag carpet appearance are varied with areas that show less microporosities. Dentine tubules were completely opened and cross-banded collagen was clearly evident at the tubule orifice walls, indicating that resin tags will definitely contribute to retention and sealing of the resin-dentine bond".
- "For the specimens with enamel that surrounds mid-coronal dentine and has been prepared with 600- grit silicon carbide paper, a highly retentive acid-etch pattern was exposed that definitely offers sufficient retention and sealing for the resin-enamel bond".

Following the application of NRC, Dentsply recommends the use of Prime&Bond NT as the adhesive. According to the manufacturer, Prime&Bond NT (PBNT) incorporates an extremely fine filler (nanofiller) to maintain a low viscosity and therefore good penetration. Because the nanofiller is so small (about 7 nm) it is capable of entering the

tubules and the demineralized zone of the dentine. It is also capable of penetrating the spaces between the collagen microfibrils of the dentine. The nanofiller also serves as an additional crosslinker and therefore strengthens the adhesive layer and the hybrid layer providing better micromechanical retention. Dentsply refers to this microretention as nanoretention (Prime&Bond NT Technical Manual, 1998).

Component	Function
PENTA	Adhesion promoter, wetting aid and crosslinker
UDMA resin	Resin molecule of intermediate elasticity
Resin R5-62-1	Elastomeric resin in the cured network
T resin	Small, crosslinking molecule
D resin	Small mobile resin for dentine infiltration
Nanofiller	Nanoscale filler for strength and crosslinking
Initiators	Initiate light curing reaction
Stabilizer	Stabilizes material during storage
Cetylamine - hydrofluoride	Fluoride release
Acetone	Solvent and carrier of resins, water displacer

Table 6. Composition of PBNT and their functions (Prime&Bond NT Technical Manual, 1998).

According to the manufacturer, PBNT consists of 2 other resins, namely D-Resin and T-Resin (table 6). The D-Resin is a small resin molecule that provides for better infiltration of the porous dentine structure. The T-Resin is a crosslinking agent that leads to a denser network of the resin matrix.

In this study two of the latest compomers, namely Dyract AP^a and F2000^b were evaluated. Compomers, like other methacrylate based materials show shrinkage following polymerization. This shrinkage is partially cancelled out by a subsequent expansion of the material following the diffusion-based reaction in the presence of water (Dyract AP Technical Manual, 1997).

The new generation of compomers are alleged to shrink much less and also expand in the mouth with the absorption of water (Dyract can take up to 3% water) and therefore has the potential to overcome/minimize the initial shrinkage due to polymerization (Grobler *et al.*, 1997). F2000 has a water uptake of 39.3 g/mm³ and the subsequent expansion may probably be responsible for its good marginal seal (F2000 Technical Report, 1998).

Shear bond strength testing methods has been extensively reviewed and several reports have attempted to standardize many of the variables. In this present study a bonding area of 3 mm² to dentine was used. However, it has been shown that smaller cross sectional area of bonded specimens produced higher bond strengths (Phrukkanon *et al.*, 1998; Pashley *et al.*, 1995). Larger specimens seem to contain more defects than smaller

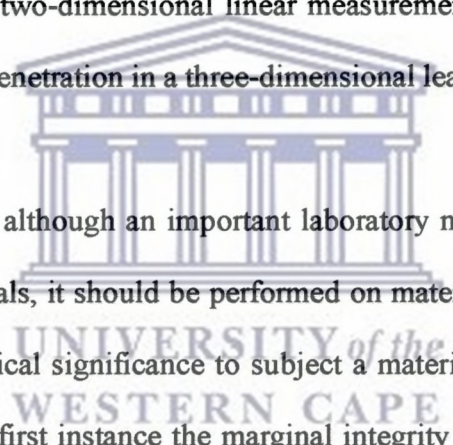
specimens and the adhesive bonding is not uniform microscopically. The interface can contain air bubbles, phase separations, surface roughness and non-uniform stress distributions. With the application of a load there is a non-uniform distribution of stresses as the first crack begins to propagate from one of the defects (Pashley *et al.*, 1995).

A new microtensile testing method by Sano *et al.* using a bonding area as small as 0.02 cm² tend to produce higher bond strengths with more interfacial bond failures than cohesive failures (Pashley *et al.*, 1995). Presumably, this new testing method provides better stress distribution at the adhesive interface and the bond is more likely to fail at its weakest point *i.e.* at the adhesive interface (Pashley & Carvalho, 1997).

Pashley *et al.* (1995) suggest new testing methods need to be developed and standardized to provide researchers with the tools needed for adhesion testing. However, the ultimate test of bond strength between materials and tooth structure is its actual performance in the oral cavity.

Similarly, microleakage studies *in-vitro* are used to test the behaviour of materials so as to help clinicians evaluate the sealing ability of these materials. The marginal integrity of dental restorations is an important parameter in evaluating the clinical success of a dental material. There are several techniques available to demonstrate *in-vitro* microleakage of restorations including radioisotopes, dyes, air pressure, neutron activation analysis, bacterial penetration, pH analysis and scanning electron microscopy. Dyes and

radioisotopes are the most commonly used. In this study the dye penetration method was used to evaluate microleakage patterns. A 2% basic fuchsin dye was used because of the simplicity of the technique as well as the ease of scoring when observed directly under a microscope. The cycling temperature range of 15° C to 45° C was selected as this was found to be the *in-vivo* temperature fluctuations on the tooth surface under normal drinking conditions (Kidd, 1976). To determine microleakage, linear measurements of dye penetration were made at the tooth/restoration interface and scored using a scale from 0 to 3. However, these are two-dimensional linear measurements and do not take into account the density of dye penetration in a three-dimensional leakage pattern.



In-vitro microleakage tests, although an important laboratory method of evaluating the clinical behaviour of materials, it should be performed on materials that create an intact seal. It would be of no clinical significance to subject a material to thermal stresses to determine leakage if in the first instance the marginal integrity was not intact. Thermal stressing of already defective leaking interfaces will only exacerbate the leakage thus giving a wrong clinical opinion. Initial leakage due to polymerization need to be differentiated from that due to thermal stresses. Thus these microleakage tests should only be performed on materials that create an intact seal as this will help determine marginal integrity following thermal stresses that occur *in-vivo* (Gale & Darvell, 1999).

Studying the micromorphological relationship between the adhesive agent and tooth structure at the interface is important as this may provide a better understanding of the

sealing ability and adhesive qualities of the bonding agent. Confocal scanning laser microscopy (CSLM) was used in this study to examine the tooth/restorative interface. The use of CSLM for the study of dental materials offers several advantages over scanning electron microscopy (SEM). CSLM enables thin optical sections to be made below the surface of intact specimens without the normal problems of thin sectioning associated with SEM or the potential drying of biological specimens (Watson, 1990). The specimens do not require any special preparation treatment and can be examined in its biological environment. Any material that has a water content will be placed under stress when prepared for SEM studies. The electron beam of the SEM requires a high-vacuum environment to function properly and materials with high water content will dehydrate and distort badly under such conditions (Ngo *et al.*, 1997). Confocal imaging has the advantage in that it allows subsurface features of the tooth/restorative interface, materials and bonds to be examined *in situ* (Watson and Boyde, 1987). This method is thus ideally suited for investigation of the penetration, fit and thickness of adhesive bonding agents as well as restorative materials (Watson, 1989).

In this study, the CSLM images of SBMP show good penetration of the adhesive into the dentine (figures 22 & 23). Resin tags are clearly seen in the dentine and measured about 100-150 μm in length. These long resin tags suggest deep penetration of the adhesive into the dentinal tubules. Acid etching with 37% phosphoric acid demineralizes the dentine and opens the tubules and subsequent rinsing off with water removes the smear layer and smear plugs present on the surface thereby enabling good penetration of the resin into the

dentinal tubules. A good hybrid layer formation indicates that there is good intermingling and enveloping of the adhesive into the collagen present on the demineralized dentine surface. The hybrid layer in this study was found to be about 5 μm in thickness. The good penetration of SBMP adhesive into the demineralized dentine, long resin tags and a distinct hybrid layer formation may all contribute to the high shear bond strength and absence of microleakage observed with SBMP in this study.

In contrast, the CSLM images of NRC and PBNT (figures 20 & 21) show much shorter resin tags. The tags measure about 10 μm in length. With this self-etching/self priming system the acid present may not demineralize the dentine to sufficient depths or open the dentinal tubules to allow good penetration of the adhesive. With the non-rinse technique the smear layer and smear plugs are not removed from the dentine surface and this may further inhibit the flow of the adhesive into the demineralized dentine. The hybrid layer was found to be much narrower measuring about 2 μm in thickness. This again suggests poor penetration of the adhesive into the collagen layer or insufficient demineralization of dentine by the NRC resulting in a much narrower hybrid layer.

Ferrari *et al.* (1999) compared the interfacial micromorphology of PBNT on conditioned and unconditioned dental substrates. In that study it was found that the hybrid layer was much narrower and the resin tags much shorter when the self-etching/self-priming NRC was used. The hybrid layer was found to be about 1-2 μm in thickness which compares favourably to this study. When the dentine surface was acid etched with 36% phosphoric

acid the hybrid layer was found to be much thicker measuring about 3-5 μm . This again suggests that the self-etching/self-primer may not demineralize the dentine to sufficient depths to allow for good penetration of the adhesive PBNT. Also the presence of the smear layer may inhibit the penetration of the adhesive into the demineralized dentine. The lack of good penetration of adhesive into the demineralized dentine and shorter resin tags may account for the lower shear bond strength and microleakage observed with NRC and PBNT in this study.



CHAPTER VI

Conclusion

From this study the following conclusions may be drawn:

- The acid-etch technique of Scotchbond Multi-Purpose Plus together with compomer F2000 produce strong bonds to tooth structure that are capable of withstanding the forces of shrinkage thereby eliminating any microleakage at the tooth/restorative interface.
- The adhesion of Non-Rinse Conditioner and Prime&Bond NT with compomer Dyract AP to tooth structure may not be strong enough to withstand the stresses of shrinkage. This was reflected by the microleakage seen with this system.
- Confocal scanning laser microscopy is a relatively new and exciting possibility for examining the relationship of dental tissue and restorative systems.
- Technologies from chemical and mechanical adhesion seem to be joining forces to produce materials with better adhesive qualities aiming at attaining one of the primary goals of restorative dentistry *i.e.* to prevent dislodgment of the restoration.



- Although laboratory testing of materials is important in evaluating and comparing new materials, no *in-vitro* test can possibly duplicate the diversity of factors in the oral environment that could affect the performance of these materials. The better the test method simulates the oral cavity conditions the more valid the extrapolation of the results. Most *in-vitro* test methods however closely they simulate the oral environment, must be best considered as necessary preliminary screening procedures to compare and access these materials.



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Appendix

- a Dentsply DeTrey GmbH, Addlestone, Surrey, UK.
- b 3M , St. Paul, Minn., USA.
- c 3M Coated Abrasives, 3M South Africa, Johannesburg, RSA.
- d Universal Polisher, Betchworth, Surrey, UK.
- e Conduit Tubing, SA PVC, Johannesburg, RSA.
- f Wild, Heerbrugg, Switzerland.
- g Johnson Controls Inc., Milwaukee, WI, USA.
- h Optilux, Demetron Research Corporation, 5 Ye Olde St., Danbury, CT., USA.
- i Cure Rite Visible Curing Light Meter, Dentsply Caulk.
- j Zwick Model 1446, Zwick GmbH & Co., Ulm, Germany.
- k SS White Inc., Lakewood, New Jersey, USA.
- l Cooley & Cooley Ltd., Houston, TX, USA.
- m Revlon, Paris, France.
- n Minitom, Streuers, Copenhagen, Denmark.
- o E. Merck, Darmstadt, Germany.
- p LSM 410, Zeiss, Germany.